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Final Report

DEVELOPMENT OF HIGH ENERGY BATTERIES

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ABSTRACT

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Research conducted on this project shows that primary battery systems with lithium anodes of sufficiently low polarizability are possible in nonaqueous electrolytes. However, the capacity of the cells developed was limited by the high polarization of the cathode, by the resistance of the solution, and by the resistance of the membrane required to prevent contact of the soluble cathode materials. Open circuit values of 3.9 volts were obtained.

Maximum current densities of (25 ma)/cm^2 have been obtained with lithium anode, propylene carbonate-KCNS-H_2O mixtures, and cathode of Halane or $KICl_4$. Under present conditions, measurements indicate maximum cell capacity of about 0.5 whr/lb. Practical values of energy per unit weight of 15-30 whr/lb seem possible with further development. At present the most promising systems are propylene carbonate-potassium thiocyanate or tetraethyl ammonium bromide mixtures with cathodes of Halane or $KICl_4$.

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DEVELOPMENT OF HIGH ENERGY BATTERIES

I INTRODUCTION

The resurgent interest in batteries has been due in part to the need for a highly portable electric power source in missiles and space probes. Electrical power is required for hydraulic motors and servo-mechanisms in guidance; for electrical equipment in telemetry, data storage, and transmission; and for signal equipment on the ground. Manned rockets will require even more extensive power for environmental control and expanded communication and guidance facilities.

Besides portability, desirable properties of batteries for power sources are the ability to withstand high inertial impulse and to operate in extreme temperatures and in any position. Primary, or one-shot, batteries are of interest because they are capable of high current drainage; they can also be used for emergency power or other temporary uses in more elaborate rockets.

In October 1959 the National Aeronautics and Space Administration authorized Stanford Research Institute to conduct a research program on a high energy nonaqueous battery. Most batteries commonly operate with 0.5-2.0 cell voltage by using moderately active anodes such as zinc, lead, or iron. Batteries utilizing more active anode materials, such as lithium or sodium, would yield higher cell potentials; thus, savings in weight and size could be realized.

Since the use of such metals with reactive solvents or water is precluded because of solvent reaction and gas evolution, the systems must be made of nonreactive materials and selected nonaqueous solvents. The nonaqueous solvent should have high dielectric constant, low viscosity, and workable liquid range--low freezing point and high boiling point. As the solvent molecule should not contain active hydrogen atoms, solvents such

as alcohols (R-OH) or acids (RCOOH) which react rapidly with alkali metals are not suitable.

The reaction metals of interest include sodium, calcium, and lithium. This research focused on lithium as anode material. The other metals would probably show similar results.

The objective in this research was the development of batteries with higher cell voltage and increased capacity (whr/lb) over that of existing batteries. By utilizing solvents with low melting points the low temperature operation of nonaqueous batteries may be improved.

II LITERATURE SURVEY

Previous work on nonaqueous batteries has been reported by T. P. Dirkse[1] related to the use of alkali or alkaline earth metals in batteries. Such solvents as liquid ammonia, hydrazine, hydrocyanic acid, ethyl alcohol, and hydrofluoric acid were discussed and studies of cell conductivities and electrolyte performance in acetonitrile, pyridine, acetone, and formamide were summarized. With the cell system sodium lithium perchloride-ethanol, silver oxide, a discharge of 80 ma/cm^2 was obtained with a cell voltage of 1.0 volt; the open circuit voltage was 3.0.

Successful batteries have been made with liquid ammonia solvent, zinc, lead and magnesium anodes, and lead dioxide, sulfur, mercury sulfate, or sulfur cathodes.[2] Experimental cells with 26 whr/lb capacity have been assembled showing open circuit voltages of 2.39 [for $(Mg/KCNS-NH_3)/HgSO_4$ cell]. Recent work[3] has suggested that a cell consisting of a magnesium anode and an m-dinitrobenzene cathode utilizing a KCNS liquid-ammonia electrolyte should result in a battery with 100 whr/lb capacity. Liquid sulfur dioxide has been used in a nonaqueous battery with sodium anodes, IBr_2, $FeCl_3$, and iron cathodes; an open circuit voltage of 5 was found.[4] Acetonitrile has been used as the solvent in a Leclanche dry cell to give 1.5 volts open circuit.[5]

III EXPERIMENTAL WORK

The major part of the present work evaluated cells made of lithium anodes and certain selected cathodes in solutions of propylene carbonate and potassium thiocyanate.

A. NONAQUEOUS SOLUTION STUDIES

1. Conductivity Measurements

Conductivity measurements were carried out with a shielded Wheatstone bridge using a modified General Radio capacitor bridge, Type No. 716-C, circuit.

A power oscillator from Industrial Test Equipment Company, Model 1040, supplied the 1,000 cps alternating current. The bridge balance point was measured visually with a Tektronix, Type 531, oscilloscope. These are shown in Fig. 1.

Conductivity measurements were made using the cell shown in Fig. 2. Caps were placed on top of the columns to prevent moisture and impurities from getting into the cell. The cell, plus 20 ml of solution, was then placed in constant temperature baths. Bath temperatures ranged from -40°C to +50°C. Conductivity measurements at various temperatures were made of saturated solutions of the salts KCl, NaCl, HBr, LiBr, Et_4NBr, and KCNS in various solvents. Propylene carbonate was studied most extensively.

Measurements were also made of saturated solutions of KCNS and Et_4NBr in dimethylsulfoxide, acetonitrile, and mixtures of N-methyl acetamide with propylene carbonate. The results of this work are tabulated in Table I.

The relation of specific conductivity, temperature, and concentration of KCNS in propylene carbonate is given in Table II and Figs. 3, 4, 5, and 6. Figures 3 and 4 illustrate how specific conductivities, K = 3.45, 6.20, 8.0, and 10.0 x 10^{-3} Ω^{-1} cm^{-1}, vary with temperature and concentration of KCNS in propylene carbonate. Figure 5 is a plot of the logarithm of the specific conductivity of KCNS in propylene carbonate versus $1/T°K$.

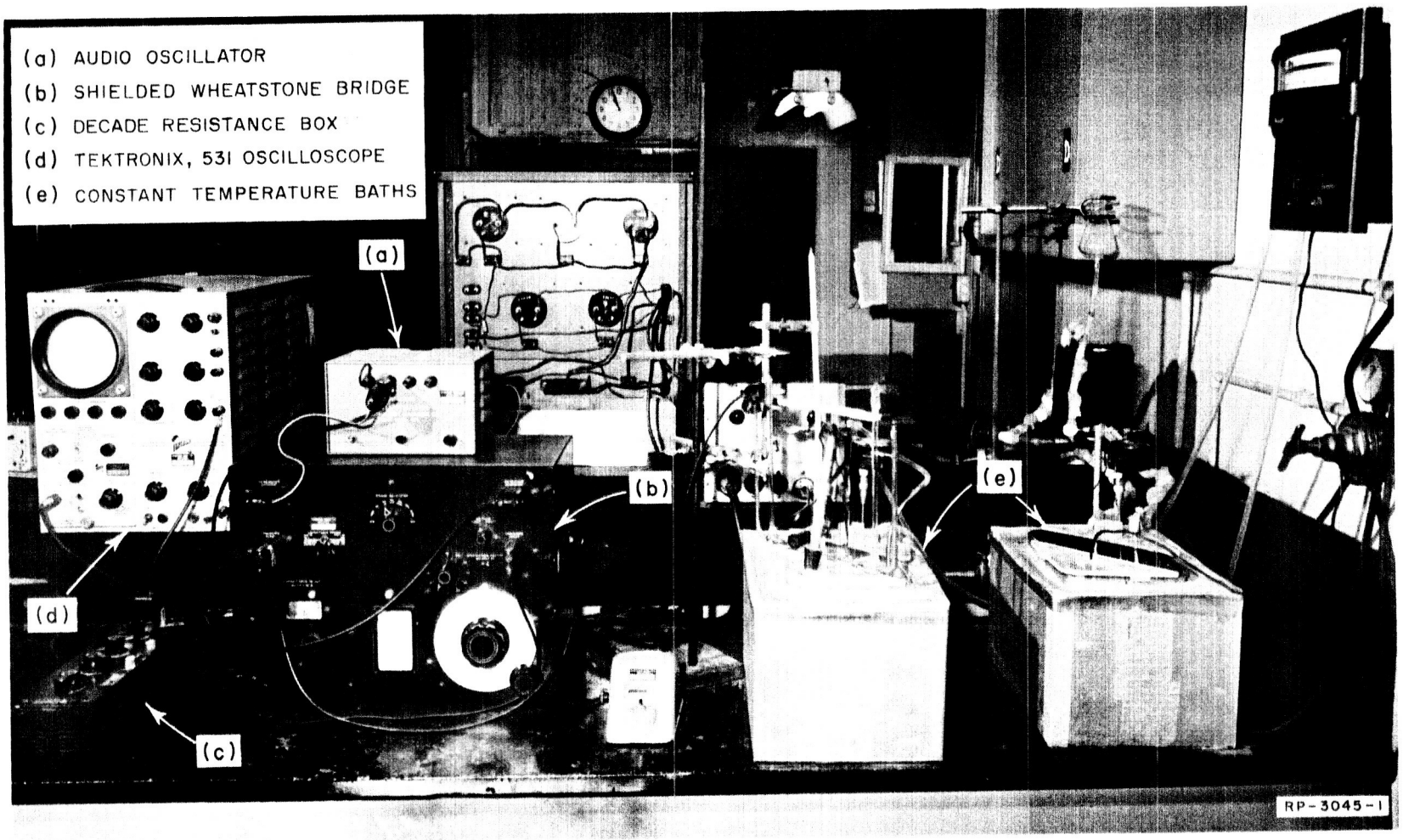

FIG. 1

CONDUCTIVITY AND CELL TESTING EQUIPMENT

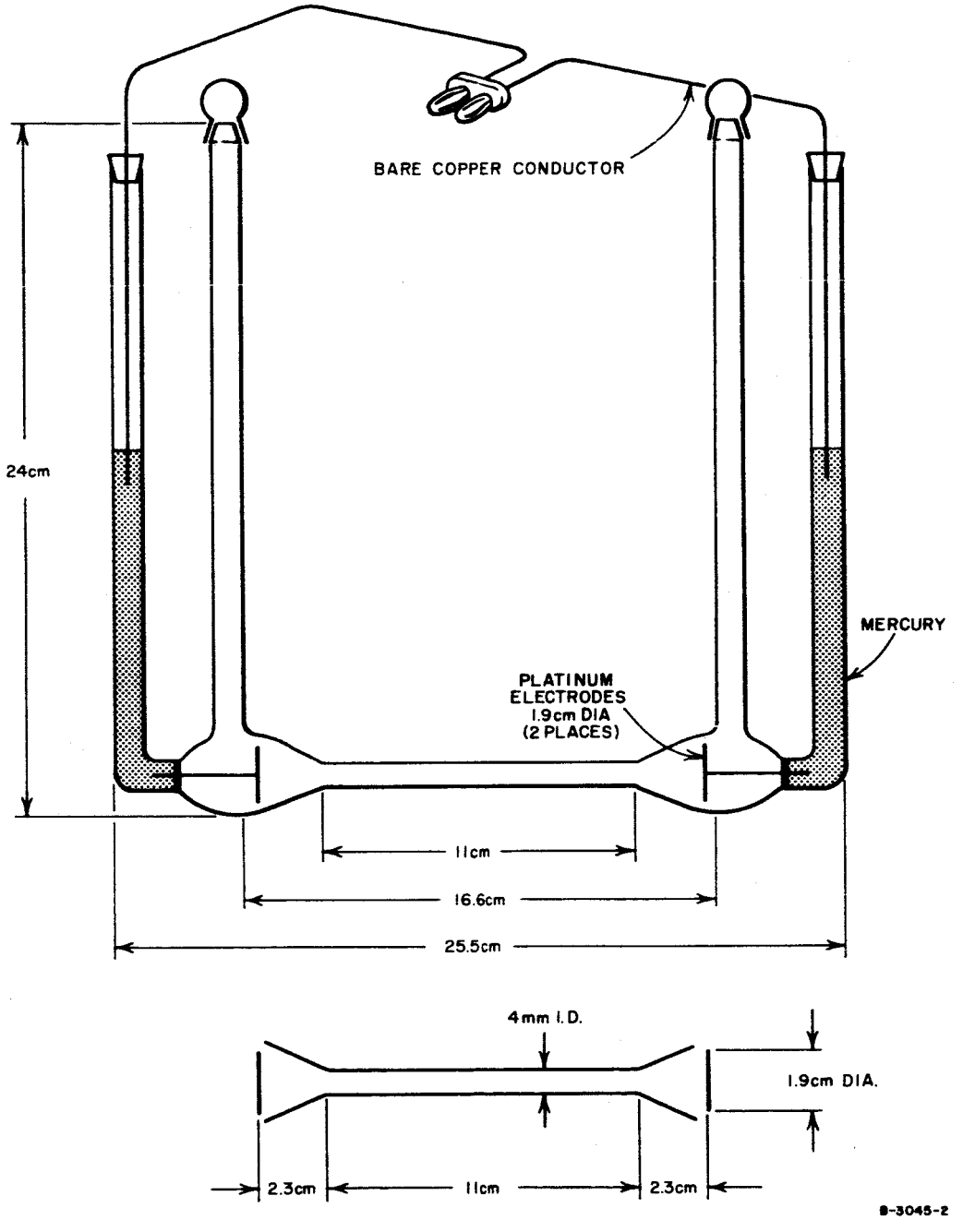

FIG. 2

CELL "A" FOR CONDUCTIVITY MEASUREMENT

Table I

CONDUCTIVITY (K) MEASUREMENTS OF VARIOUS SOLVENTS AND ELECTROLYTES AT VARIED TEMPERATURES

Run Number	Solvent (50 ml)	Electrolyte	K, Ω^{-1} cm^{-1}	T°C
1	Propylene carbonate (distilled)	None	1.8×10^{-5}	47
2		KBr saturated	1.8×10^{-4}	20
			3.15×10^{-4}	50
3		KBr 10 gm	3.13×10^{-4}	45.2
			3.09×10^{-4}	43
4		KCNS 10 gm, not saturated	8.4×10^{-3}	33
			7.09×10^{-3}	26
			7.08×10^{-3}	25.5
			1.24×10^{-2}	50
5		KCNS 11 gm, saturated	6.82×10^{-3}	25.5
			7.95×10^{-3}	31.0
			12.58×10^{-3}	50
			10.00×10^{-3}	40
6		LiBr 25 gm, saturated	4.2×10^{-4}	22.2
			5.68×10^{-4}	30
			11.45×10^{-4}	40
			15.55×10^{-4}	50
7		Solution No. 6 diluted 1 to 1 with propylene carbonate	1.355×10^{-3}	22
			1.483×10^{-3}	25
			1.853×10^{-3}	30
			2.572×10^{-3}	40
			3.50×10^{-3}	50
8		LiCl 10 gm, saturated	3.07×10^{-4}	25.2
			3.31×10^{-4}	31
			3.93×10^{-4}	40.8
			4.79×10^{-4}	50
9		Et_4NBr 10 gm saturated	7.039×10^{-3}	24.2
			7.77×10^{-3}	30
			9.417×10^{-3}	40.3
			11.09×10^{-3}	50
10	Propylene carbonate (as received)	H_2O 0% (added)	6×10^{-3}	25
		H_2O 3.8%	1.25×10^{-2}	25
		H_2O 10%	2.20×10^{-2}	25
		H_2O 20%	3.82×10^{-2}	25

Table I (Concluded)

Run Number	Solvent (50 ml)	Electrolyte	K, Ω^{-1} cm^{-1}	T°C
11	Dimethyl sulfoxide	KCNS 10 gm, all dissolved	1.292×10^{-2}	22
			1.396×10^{-2}	25
			1.578×10^{-2}	30
			1.993×10^{-2}	40
			2.458×10^{-2}	50
12		Et_4NBr 8 gm, saturated	1.035×10^{-2}	24
			1.170	30
			1.431	30
			1.713	50
13		KCNS 11 gm, unsaturated	1.225×10^{-2}	22.8
			1.309	25
			1.516	30
			1.937	40
			2.492	50
14	Acetonitrile	Et_4NBr 8 gm, saturated	3.40×10^{-2}	50
			3.17	40
			2.94	30
			2.81	25
15		KCNS 10 gm, saturated	2.68×10^{-2}	23
			2.73	25
			2.84	30
			3.07	40
			3.30	50
16	111.90 gm propylene carbonate 92.85 gm N methyl acetamide	KCNS 10 gm, unsaturated	6.57×10^{-3}	25
			7.67	30
			6.39	25
			7.66	30
			10.48	40
			13.30	50

Table II

RELATION OF SPECIFIC CONDUCTIVITY, TEMPERATURE, AND CONCENTRATION OF KCNS IN PROPYLENE CARBONATE

Moles KCNS	Cell No.	Constant	K = C/R	0.0°C	20.0	25.0	30.0	35.0	40.0
1.95	2	120.5	R	42,210	20,030	17,510	15,310	13,510	11,990
			K	2.86 x 10^{-3}	6.02	6.88	7.88	8.92	10.01
1.76	1	122.3	R	40,640	20,100	17,460	15,320	13,580	12,110
			K	3.01 x	6.09	7.02	7.99	9.03	10.02
1.55	2	120.5	R	28,700	15,230	13,470	11,970	10,710	9,642
			K	4.20	7.91	8.95	10.08	11.23	12.50
1.285	1	122.3	R	35,180	18,870	16,630	14,790	13,250	11,950
			K	3.48	6.49	7.36	8.28	9.23	10.23
0.961	2	120.5	R	33,120	18,480*	16,620	14,900	13,460	12,230
			K	3.64	6.53	7.25	8.09	8.95	9.85
0.648	1	122.3	R	35,470	20,690*	18,760	16,910	15,390	14,040
			K	3.45	5.91	6.53	7.23	7.95	8.71
0.305	1	122.3	R	45,760	28,120	25,500	23,220	21,250	19,550
			K	2.68	4.35	4.80	5.27	5.76	6.26
$1/T \times 10^3$				3.66	3.41	3.36	3.30	3.25	3.19

* 20.4°C

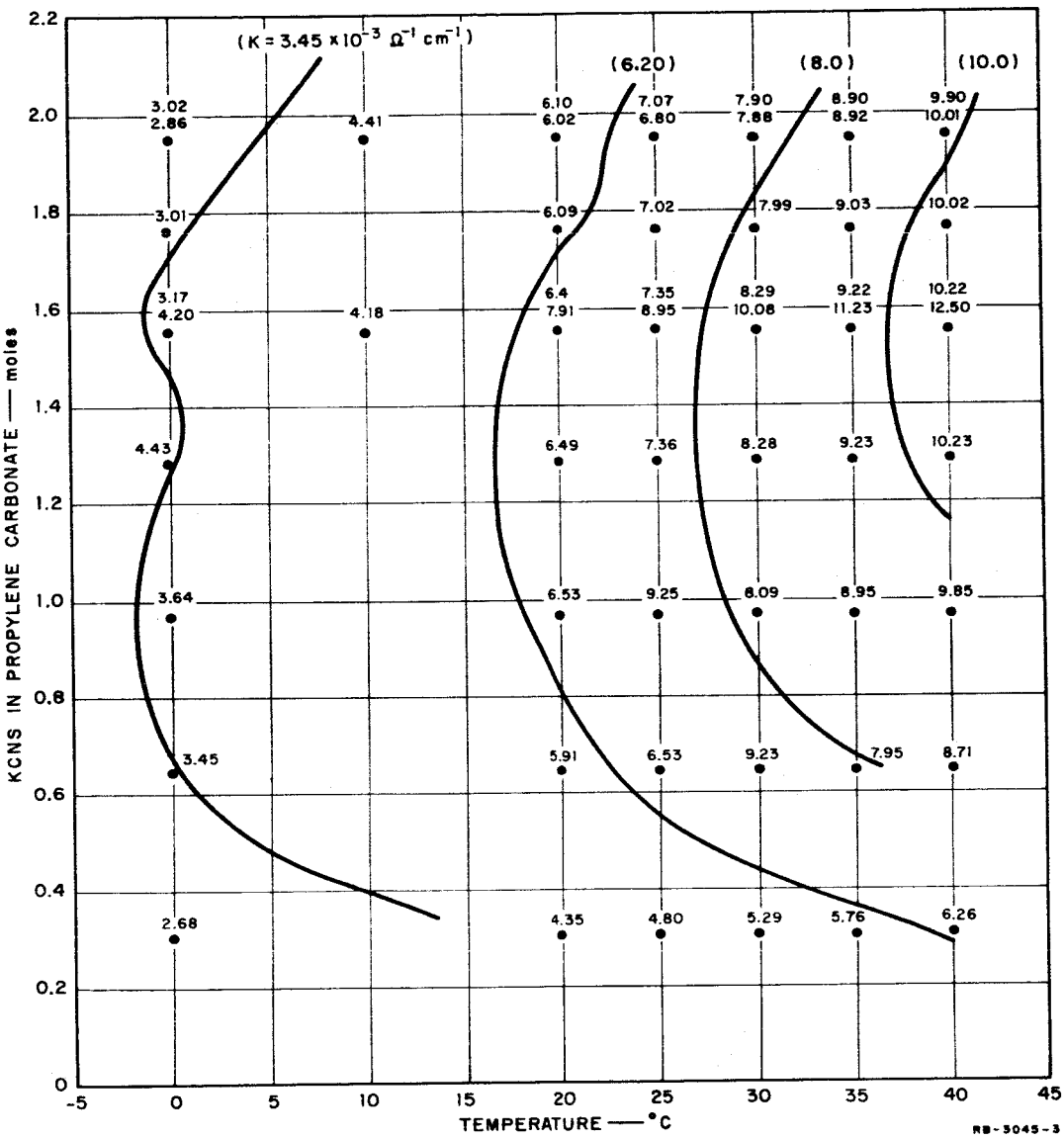

FIG. 3

RELATION OF SPECIFIC CONDUCTIVITY TEMPERATURE AND CONCENTRATION OF KCNS IN PROPYLENE CARBONATE

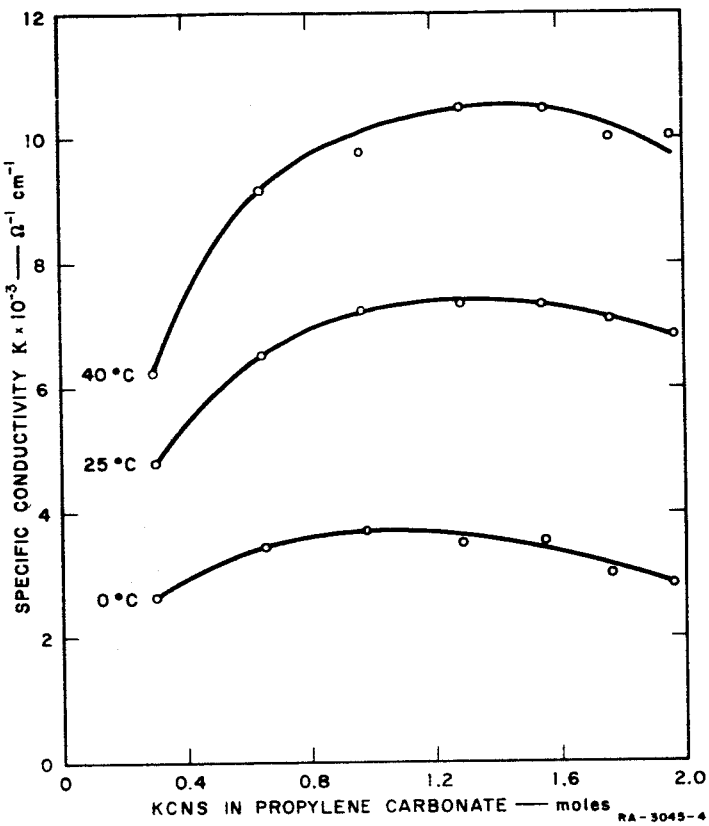

FIG. 4

VARIATION OF SPECIFIC CONDUCTIVITY IN PROPYLENE CARBONATE-KCNS WITH TEMPERATURE AND CONCENTRATION

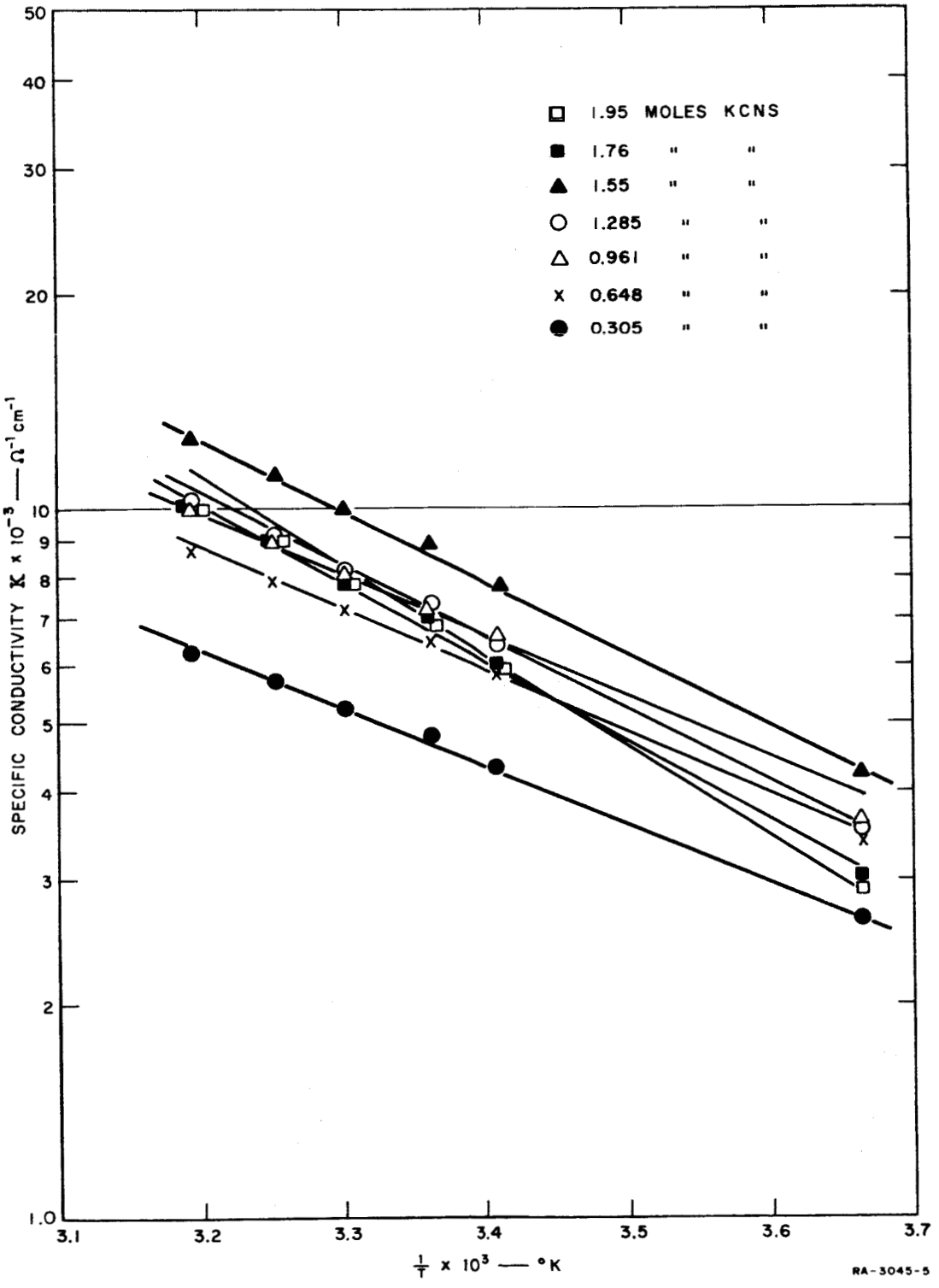

FIG. 5

LOGARITHM OF THE SPECIFIC CONDUCTIVITY OF KCNS IN PROPYLENE CARBONATE vs 1/T°K

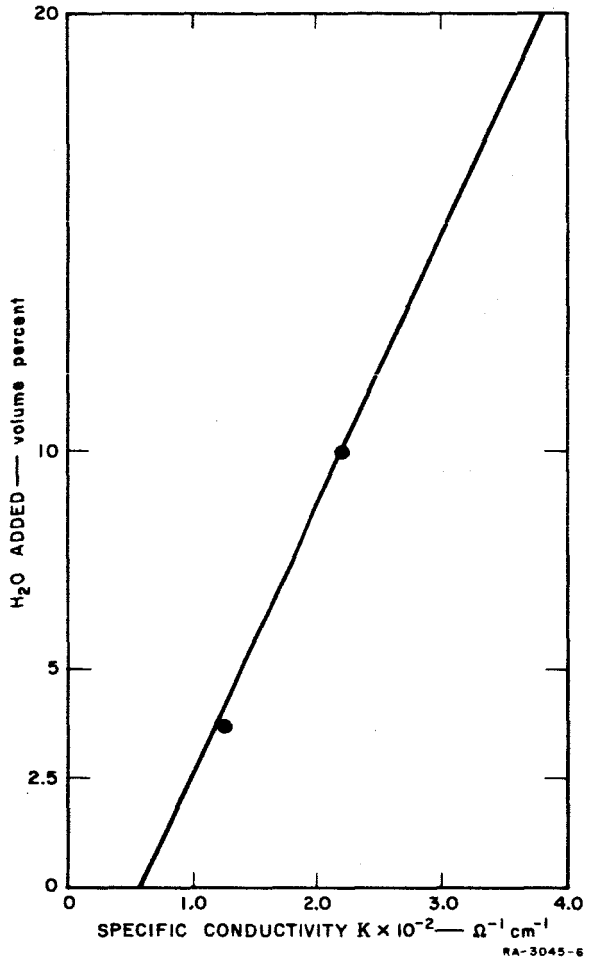

FIG. 6

SPECIFIC CONDUCTIVITY OF PROPYLENE CARBONATE—POTASSIUM THIOCYANATE SOLUTIONS WITH H_2O ADDED

Figure 6 illustrates how the specific conductivity of KCNS in as-received propylene carbonate changes with water content at 25°C.

2. Compatibility with Lithium

Nonaqueous solvents were surveyed and those of interest are tabulated with their various bulk properties in Table III. The compatibility of these as-received and purified liquids with lithium has been ascertained. These results are shown in Table IV. Commercial solvents are available which are compatible with lithium metal and which have high conductivity with electrolytes; thus they could be used in battery systems. Neither

Table III

PHYSICAL CONSTANTS OF NONAQUEOUS SOLVENTS

Solvent	ϵ	ν^{25t} (cp)	$\frac{\epsilon}{\nu}$ (cp)	F. P. °C	B. P. °C	Molecular Weight	Source	Grade	Density
Acetaldehyde	21.1	0.21	104	-124	20	44.23	Eastman Organic Chemicals	Commercial	0.783
Acetonitrile	37.5	0.39	96.3	-41	82	41.05	Fisher	0.01% H_2O	0.783
Water	78.4	0.897	88	0	100	18		Distilled	0.998
Acetone	20.47	3.040	6.73	-95	56.5	58.08	Fisher	C.P.	0.792
N Methyl Acetamide	165.5	0.0302	5,517	28	206	73.09	Eastman Organic Chemicals	Commercial	0.942
Ethylene Carbonate	89.12	2.547	34.94	36.4	248	88.03	Matheson, Coleman and Bell	Commercial	1.338
Nitroethane	28.06	0.66	42.51	-90	114.8	75.07	Eastman Organic Chemicals	Commercial	1.052
Dimethyl Formamide	36.71	0.802	45.89	-61	153	73.097	Fisher	Certified Reagent	0.944
γ Butyrolactone	39.1	1.75	22.34	-43.5	204	86.09	Eastman Organic Chemicals	Commercial	1.125
Dimethyl Sulfoxide	45	1.98	22.72	18.45	189	78	Crown Zellerbach	Commercial	1.100
Propylene Carbonate	64.4	2.53	25.45	-49.2	241.7	102.04	Matheson, Coleman and Bell	High Purity	1.198

Table IV

COMPATIBILITY OF SOLVENTS WITH LITHIUM METAL

SOLVENT	EXPOSURE TIME--4HOURS				EXPOSURE TIME--24 HOURS				EXPOSURE TIME--3 WEEKS			
	Gas	Color of Liquid	Precipitate	Condition of Metal	Gas	Color of Liquid	Precipitate	Condition of Metal	Gas	Color of Liquid	Precipitate	Condition of Metal
Propylene Carbonate	Slow reaction	Clear	Slight in bottom	Bright		Same as at 4 hours exposure			--	Clear	White on bottom	Gray. No shiny area left on surface
γ Butyrolactone	Slow reaction	Clear	Slight in bottom	Bright	--	Clear		Partially covered with white coat	Slow reaction	Clear	White on bottom	White and black coat on surface
Dimethyl Formamide	Slow reaction	Clear for 5 min Yellow in 4 hr	Slight	Bright	--	Yellow	Buff colored	Flocculent buff around metal	All reacted	Yellow-white precipitate on top--orange layer on bottom		
Dimethyl Sulfoxide	Slow reaction	Slight yellow	Slight	Dull	Still reacting	Bright yellow	--	Black film	Slow reaction	Yellow	White on bottom	Gray coating
Nitroethane	Slow reaction	Clear	White	Dull in 2 hr	Still reacting	Clear	White on bottom	White around lithium	--	Clear	Yellow-white	Yellow-white on top
Ethyl Sulfate	Slow reaction	Cloudy	White	White coating	Still reacting	Clear	White	White	Slow reaction	Clear	White on bottom	White
Ethyl Cyanoacetate	Gassing	Yellow	White	Bright	--	Orange		All reacted	--	Red-brown	In suspension	All reacted
Acetonitrile	Gassing	Very yellow	Yellow near lithium	Coated	--	Yellow	Orange	All reacted		Orange	Orange	
2 Furaldehyde	Gassing	Dark brown	--	--	--	Black				Black		All reacted
Butanol	--	Clear	White	White coating	--	Light yellow	White on bottom	All reacted		Light yellow	White on bottom	
Isopropanol	--	Clear	White	White coating	--	Clear	Large white flocculent	All reacted				
Formamide	--	Pale yellow	White on bottom	All reacted	Faint NH_3 odor							
Propargyl Alcohol	Violent gassing with heating	Placed in hood and solvent removed in 2 min										

acids nor ammonium ions were acceptable with lithium, since rapid corrosion occurs. Therefore, salts such as KCNS and Et_4NBr must be used as electrolytes.

Propylene carbonate used in this study originated from Matheson, Coleman, and Bell. (Its properties and general characteristics are listed in Table III.) Raw or as-received propylene carbonate contains 2.6% water when analyzed by the Karl Fisher titration.

3. Purification of Propylene Carbonate

In order to purify the propylene carbonate a fractional distillation apparatus, shown in Fig. 7, was used. Cuts of 30 ml each were taken; the first two cuts were discarded. Conductivity measurements were made at 25°C on succeeding cuts using a 1,000 cycle AC bridge. The original propylene carbonate (as received) had a specific conductivity, $K = 1.1 \times 10^{-5}\ \Omega^{-1}\ cm^{-1}$, and the third cut of distillate had a $K = 3 \times 10^{-7}\ \Omega^{-1}\ cm^{-1}$. The fifth cut had a $K = 2 \times 10^{-7}\ \Omega^{-1}\ cm^{-1}$. Extractions of $K = 6 \times 10^{-5}$ to $8 \times 10^{-5}\ \Omega^{-1}\ cm^{-1}$ were used in cell runs.

A one-liter flask was adapted with a Nichrome heater in the bottom to heat the propylene carbonate. A reflux column 1-1/8 inch ID, 40 inches long was filled with Pyrex glass helices 3/16 inch ID (Scientific Glass Apparatus Company). The tube was wrapped with heating tape (manufactured by Electrothermal Engineering Company, Limited, and sold in the United States by Arthur S. LaPine and Company in Chicago, Illinois). This was done to heat the column sufficiently to permit the vapors to reach the condenser and collect in a calibrated flask. The system was then closed off and 20-30 ml of purified propylene carbonate was delivered into the collection flask, removed, and replaced with a new flask placed in line. An outer tube 2-3/4 inches ID was placed over the wrapped reflux tube to maintain the inside temperature.

The liquid and reflux column were kept at 126° and 110°C respectively. The entire system was maintained at 10 mm Hg.

B. ANODE PREPARATION

Lithium was selected as the anode material. Lithium has a low equivalent weight (6.9 equivalents/mole) and would be an ideal electrode

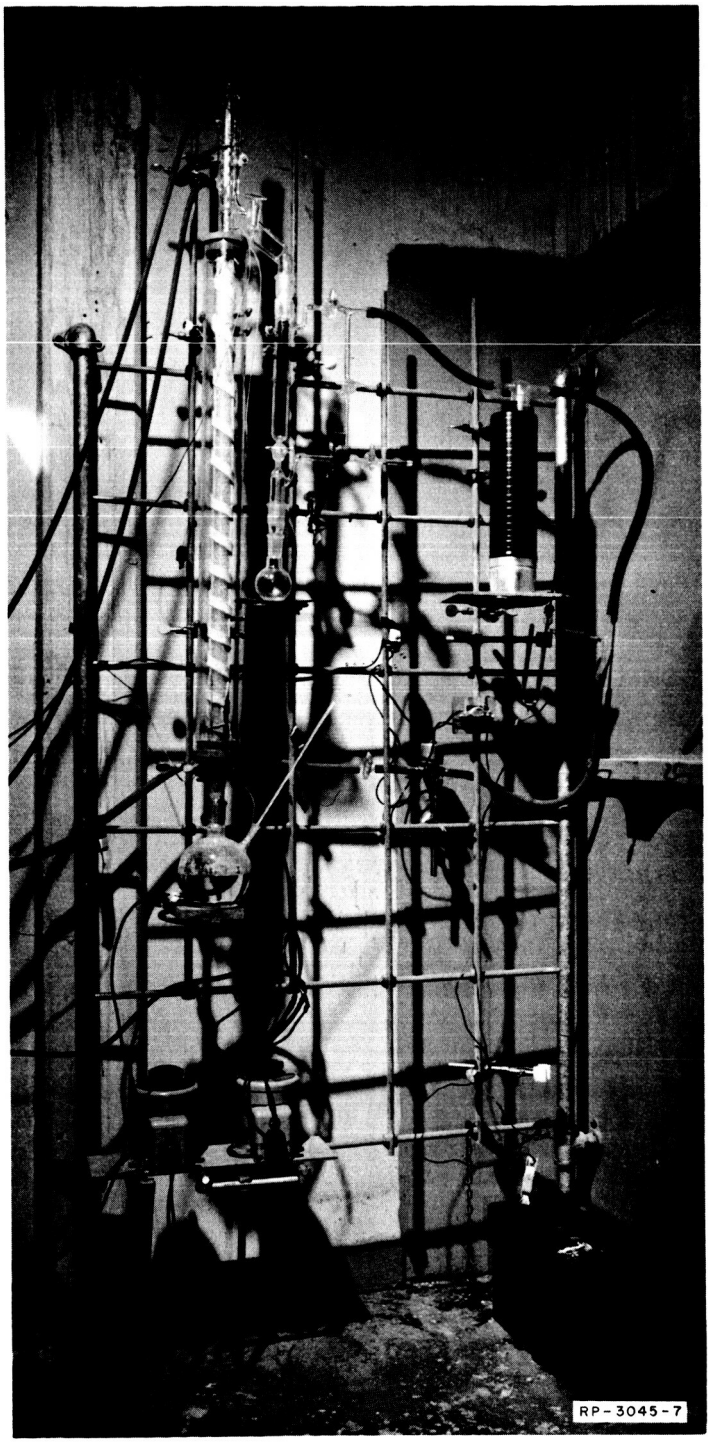

FIG. 7

DISTILLATION APPARATUS FOR PROPYLENE CARBONATE

material from the standpoint of lightness in weight and high voltage, based on its half reaction to produce lithium ion.

$$Li \rightarrow Li^{+}_{(Aq)} + e \qquad E^{\circ} = 3.05 \text{ volts}$$

The potential for the half reaction in solvents other than water (such as propylene carbonate) is expected to be in the same range, 2.8 to 3.2 volts. This voltage range is found for methanol, acetonitrile, formic acid, and ammonia.[6]

For a reaction in water between lithium and chlorine to produce solid LiCl

$$1/2\ Cl_2 + Li \rightarrow LiCl_{(c)}$$

the standard free energy change is -91.7 Kcal/mole, which is equivalent to a potential of 4.2 volts. Three methods of anode preparation were selected; use of lithium strips, lithium dip coatings, and electrodeposition of lithium.

1. Lithium Strips

Lithium in 30-mil-thick strips is soft and must be supported when used as an electrode. The first method used to form an electrode was to attach 1/4-inch-wide x 30-mil-thick lithium ribbon to copper plates. The lithium, stored in petroleum grease, was cleaned according to the following procedure:

(a) Degrease in acetone, wipe with paper tissue

(b) Soak in a methanol-dry ice mixture until the gray oxide-nitride coating is dissolved

(c) Remove from mixture, dry with paper tissue, and place in a cell prefilled with electrolyte solution.

This method gave poor mechanical attachment of lithium to copper, and showed difficulty of maintaining good electrical contact.

2. Dip Coating

A copper sheet (cut to size) was dipped into molten lithium under argon in a dry box. The lithium electrode was then assembled in the

battery cell in the dry box (Fig. 8). Covers were applied to the cell, which was then removed from the dry box for filling with electrolyte solution and testing. Lithium-coated copper anodes dipped into propylene carbonate solutions were found clean and shiny for a sufficient time to effect an air transfer to cell. Cells have also been assembled at test equipment location.

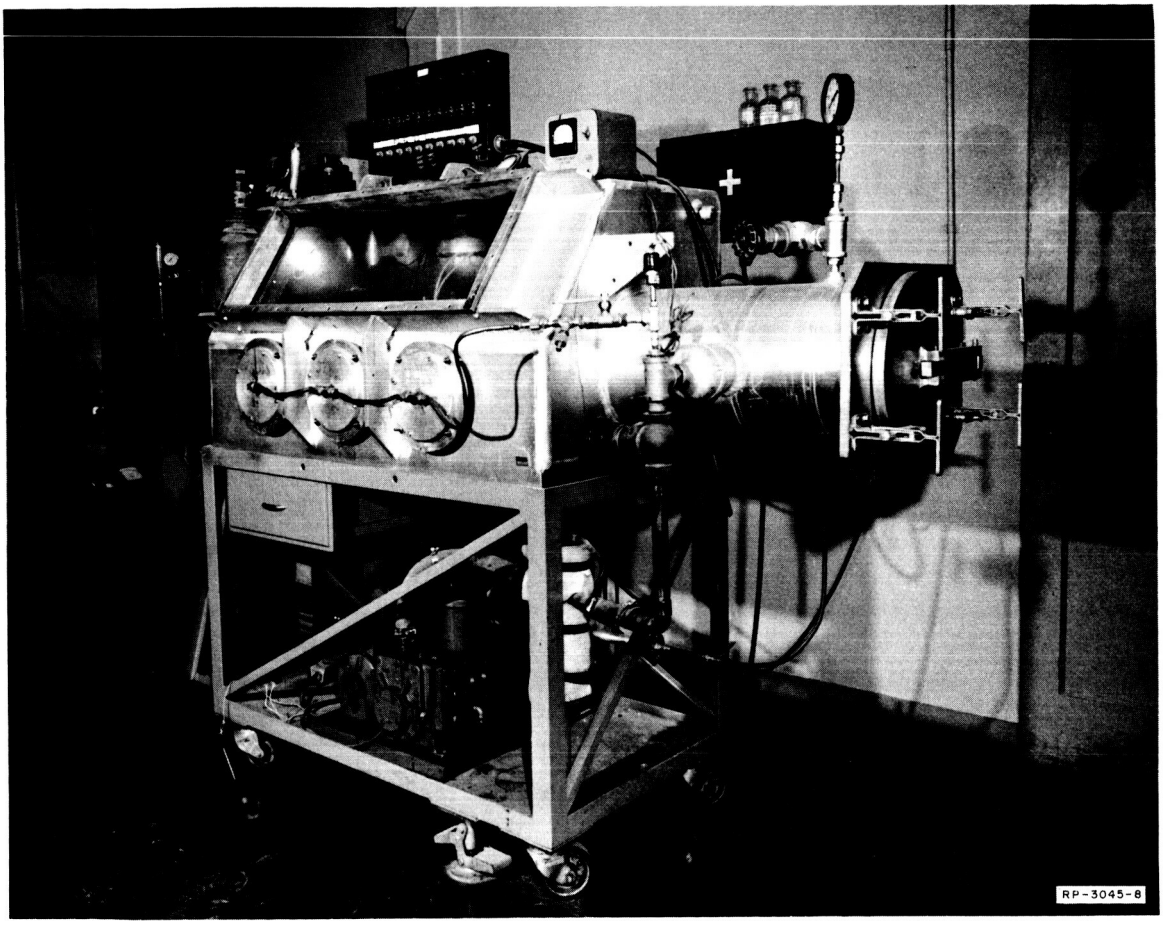

FIG. 8
DRY BOX

Some copper was dissolved by the molten lithium. In one typical run with a lithium coating of 0.31 gram spread over a 22 cm^2 surface, 0.11 gram copper was dissolved in the lithium coating and 0.29 gram of copper was left in the molten bath. After the copper had been dipped

into the molten lithium bath a gray to black colored film was formed on the surface of the bath which was skimmed off. Occasionally a voluminous black efflorescence occurred at the surface in the stainless steel pot and the black material overflowed the pot, necessitating the replaqement of the lithium.

Because the copper was soluble in molten lithium, different materials were also used. For example, molybdenum sheet was cut to desired size and then dip-coated with lithium, making an electrode which was not soluble in the molten lithium.

3. Electrodeposition

Electrodeposition of lithium onto copper plates for use as anodes was carried out in propylene carbonate solutions of lithium salts. Solutions with lithium chloride and lithium bromide produced gray crystalline deposits on the copper plates. Preliminary experiments gave current efficiencies of 20% to 40%. Electrodeposition of lithium on a metallic backing was a possible method for cell assembly; however, in the majority of the work reported, dip-coated electrodes were used since considerably more development was necessary for electrodeposition.

C. CATHODE PREPARATION

Proper selection of the cathode material was of major importance in this work. Many materials having a good charge-to-weight ratio were not compatible with solvents. For example, if chlorine gas were used the half reaction would be

$$1/2\ Cl_2 \rightarrow Cl^-_{(Aq)} - e \qquad E^\circ = +1.36 \text{ volts} .$$

Chlorine, however, decomposes solvents such as propylene carbonate.

For a more suitable cathode material such as Halane (NN dichloro-dimethyl-hydantoin) the potential would be less. In propylene carbonate solutions, Halane liberated bromine and iodine from bromide and iodide salts. Chlorine from chloride ion was not observed under these conditions.

$$(\text{Halane}) \rightarrow (\text{Halane})^= = -2e \qquad E \sim 1.0 \text{ volt}$$

or

$$RNCl \rightarrow RN^{-} + Cl^{-} - 2e \ .$$

Halane was used as a cathode material in most cell experiments. Potassium thiocyanate, since it was soluble in solvents and compatible with lithium anodes, was the electrolyte used in most cell experiments. However, Halane reacts with the thiocyanate ion to form colorless thiocyanogen. Thiocyanogen subsequently polymerizes to form a yellow-orange, unreactive compound parathiocyanogen. Alternatively, the thiocyanogen could react at the cathode.

$$1/2\ (CNS)_2 \rightarrow CNS^{-} - e \qquad E^{\circ} = +0.77 \text{ volts}$$

Formation of thiocyanogen and parathiocyanogen by removal of thiocyanate may account for the increase in resistance which occurred in many of the cell experiments. Initially the cathode was made of copper strips or wire screen covered by dipping into a molten graphite-Halane mixture. Halane can be melted without decomposition.

In later experiments the cathode was constructed using porous carbon plates permeated with the graphite-Halane mixture. Other cathode materials include copper oxide-graphite mixture, cuprous sulfide, Dibromantin-graphite, MnO_2, and CuO. Soluble copper salts such as CuF_2 and $CuCl_2$ have been mixed with graphite and placed on graphite plates in a paste form. Sulfur-graphite, potassium cyanide, silver peroxide, and silver metal on silver mesh cathode have also been tried. Potassium tetrachloroiodide ($KICl_4$) was prepared in the laboratory for use as a cathode. It is possible that this compound may form inter-Halogen-like compounds with the thiocyanates or thiocyanogen in the cell. Compound preparation of $KICl_4$ and Cu_2S are given in Appendix A. Table V contains the molecular weights, the number of electrons accepted during reaction, and the theoretical watt hours/lb calculated from a lithium cell using a given cathode material. Methods of cathode preparation are summarized in Appendix A.

Table V

CATHODE MATERIALS

Material	Molecular Weight	Electron Change	Vendor	Theoretical Cell Capacity Lithium-Cathode Material (watt hr/lb)
Halane	197	4	Jefferson Chemical Company	872
Dibromantin	296	4	Arapahoe Chemical, Incorporated	527
CuO	79	2	J. T. Baker AR	915
$CuCl_2$	135	2	J. T. Baker AR	586
O_2	32	4		2,760
S	32	2	Braun-Knecht-Heimann Company, N.F. grade	1,220
Ag_2O_2	232	2	City Chemical Company, New York	300
$KICl_4$	307	3	Laboratory preparation--Appendix	392
Cu_2S	159	2	Laboratory preparation--Appendix	290

D. MEMBRANES OR DIAPHRAGMS

Initially diaphragms were made of three to six layers of Whatman No. 1 filter paper. Although this material had low electrical resistance in solution, the separation of the anode and cathode members was not satisfactory and final work was conducted using other membrane materials. Pyroxylin membranes are solubilized by propylene carbonate. Polyvinyl alcohol films and an ion exchange membrane Nalfilm No. 1 are slowly attacked by propylene carbonate and tear while swelling. Cellophane films are swollen slightly by propylene carbonate and give too high a cell resistance for use. An ion exchange membrane with resin reinforced by Fiberglas mesh Nepton CR61, obtained from Ionics Incorporated, is usable for runs of short duration. Solvent resistance of membrane materials are presented in Table VI. A microporous polyvinyl chloride, "Pormax," manufactured by the Electric Storage Battery Company was used in the last experiments and was found to have good properties.

Table VI

MEMBRANE MATERIALS

Material	Solvent	Results	Time Immersed
Polyvinyl Alcohol	Acetonitrile	No attack	20 days
	Acetone (Tech)	No attack	20 days
	Propylene carbonate	No attack	20 days
	γ Butyrolactone	Slow attack	20 days
	DiMethyl sulfoxide	Completely dissolved	2 hours
	DiMethyl Formamide	Very slow attack	20 days
	Nitroethane	Very slow attack	20 days
Cellophane	(No attack in above solvents after 30 days.)		
Nalfilm D-30	Propylene carbonate	Dissolves readily	
Ionics Exchange Resin Nepton CR61	Propylene carbonate KCNS	No attack	30 days
Pyroxylin	Propylene carbonate	Dissolves readily	2 days
Ion Exchange Membrane Nalfilm I	Propylene carbonate	Tears while swelling	2 hours
Pormax Micro Porous-Polyvinyl Chloride	Propylene carbonate	No attack	20 days

E. BATTERY STUDIES

1. Cell Construction

Cells were constructed from 1/4-inch-thick polyethylene with inner dimensions of 1/2 x 1-3/8 x 4-3/8 inches. Cells were used with both 1/2-inch-wide electrodes and 2, 4, 5, and 9-mm spacings between electrodes, and with 1-3/8-inch-wide electrodes and 0.5- to 3-mm spacings. These were cell types I and II respectively, and are shown in Fig. 9.

Lucite was dissolved by propylene carbonate and thus was ruled out as a structural material. Polyethylene is inert to propylene carbonate and was, therefore, used in the cell design. The anode of the cells was made of lithium strips backed with aluminum or copper for support. For later experiments it was decided to use a more inert or insoluble material for support. When lithium strips were used, bakelite (1/32 inch thick) was employed for support. When used in cast form, copper or molybdenum sheet was used as the supporting member.

Multiple or bipolar (Type IV) cells have been constructed for insertion into the standard polyethylene cases, described as follows:

(a) Two copper sheets coated with Acheson Colloids "Aldag No. 154" on one side only--two coats, with a 30-minute bake at 100°C between coats and after second coat

(b) One copper sheet coated on both surfaces with "Aldag No. 154"--two coats with a 30-minute bake at 100°C between coats and after second coat

(c) The two copper sheets with one side coated, dipped into molten lithium

(d) "U"-shaped polyethylene spacers 1/4 inch thick were cemented with rubber cement, Li surface and dag surface. A compressed Halane, KBr, graphite cathode was placed in compartment between two electrodes in each compartment.

(e) Two sheets (cut to size) of filter paper placed between cathode and lithium surface

(f) The assembly is placed in outer cell and electrolyte placed in the two compartments. A drawing of the multiple cell is given in Fig. 10.

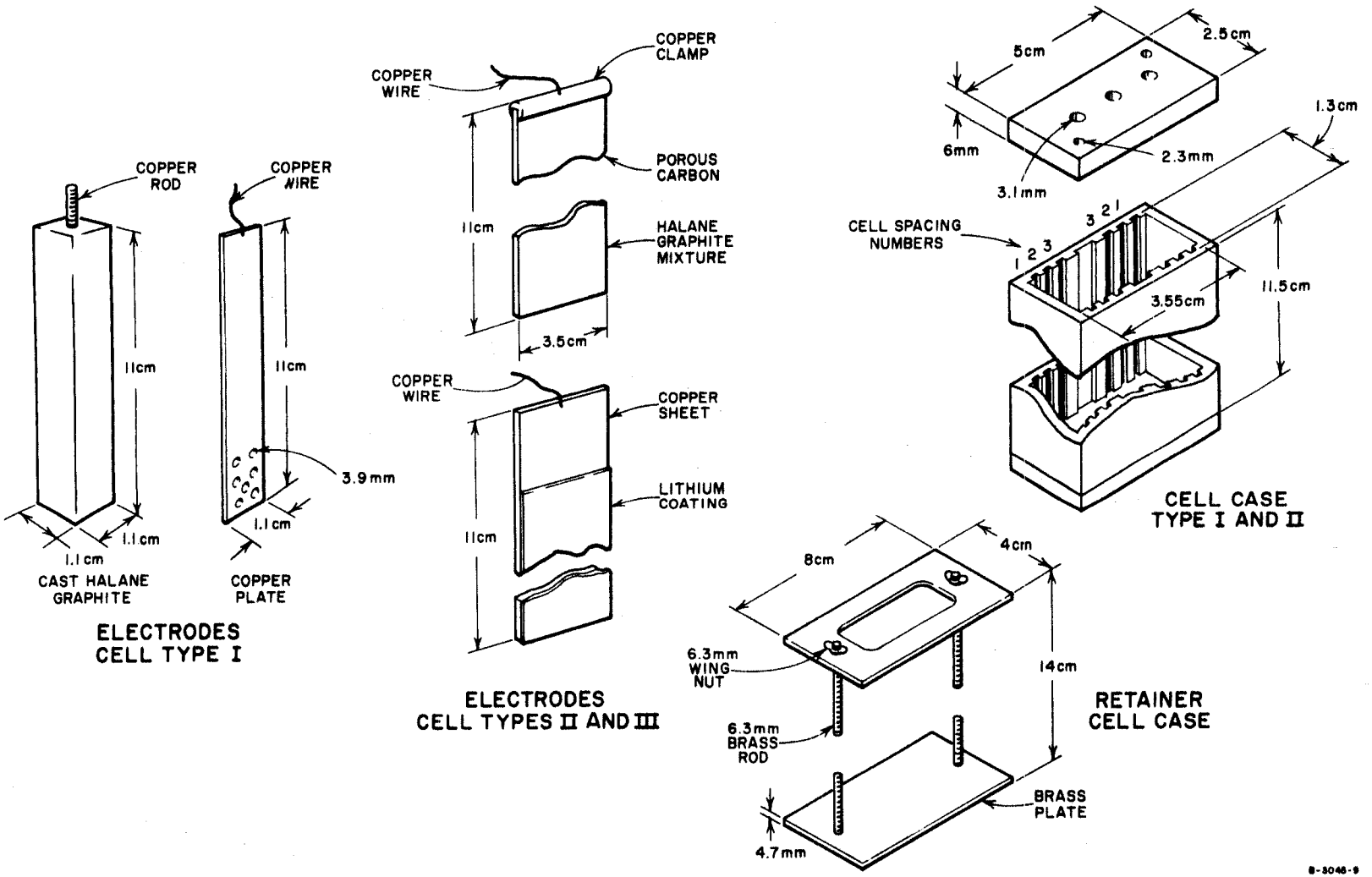

FIG. 9

DIAGRAM OF CELL APPARATUS

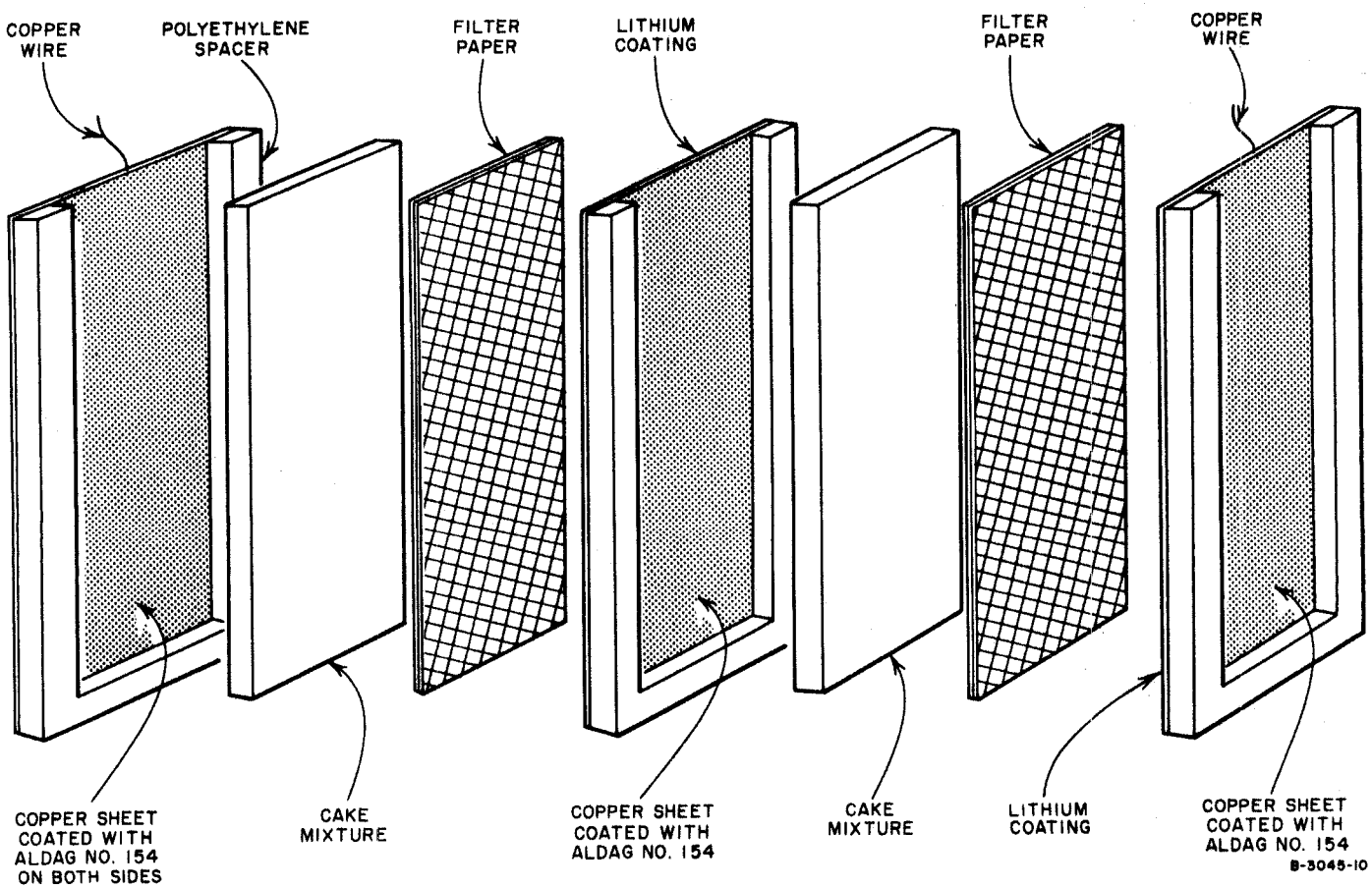

FIG. 10

BIPOLAR CELL—TYPE IV
(Placed in Standard Battery Case)

2. Cell Testing

Cells were assembled and filled with electrolyte just prior to testing. Cell types I and II required 30-35 ml of electrolyte; Cell III, 25-30 ml; and Cell IV, 6-8 ml. Measurements were made of the open circuit voltage, maximum current, and current capacities under different loads. The equipment used in these tests is illustrated in Figs. 1 and 11. The results of this work are presented in Tables VII, VIII and in Appendix B. The resistance of the cells was measured using an Industrial Equipment Company power oscillator, Model 1040, (a) to supply the 1,000 cps alternating current and a shielded Wheatstone bridge circuit (b) in conjunction with a decade resistance box (c).

Table VII

CURRENT CAPACITY OF HALANE CELLS

Cell Run (Number)	Cathode (Halane)	Capacity (ma min)	Current (ma)	Membrane	Voltage	OCV	Cell Resistance (ohms)
80	2 gms	270	10	Ion Exchange	3.05	3.60	16 At finish
79	2 gms	50	50	Ion Exchange	1.20	3.40	
78	2 gms	260	10	Filter Paper	3.30	3.50	
77	2 gms	50	50	Filter Paper	2.02	3.60	

Table VIII

CURRENT CAPACITY OF $KICl_4$ CELLS

Cell Run (Number)	Cathode ($KICl_4$)	Capacity (ma min)	Current (ma)	Membrane	Voltage	OCV	Cell Resistance (ohms)
82	2 gms	1050	10	Filter Paper	3.40	3.55	5.82 Initial
86	2 gms	1250	50	Filter Paper	3.05	3.45	7.42 Initial 21.5 At finish
81	2 gms	1100	100	Filter Paper	2.70	3.62	

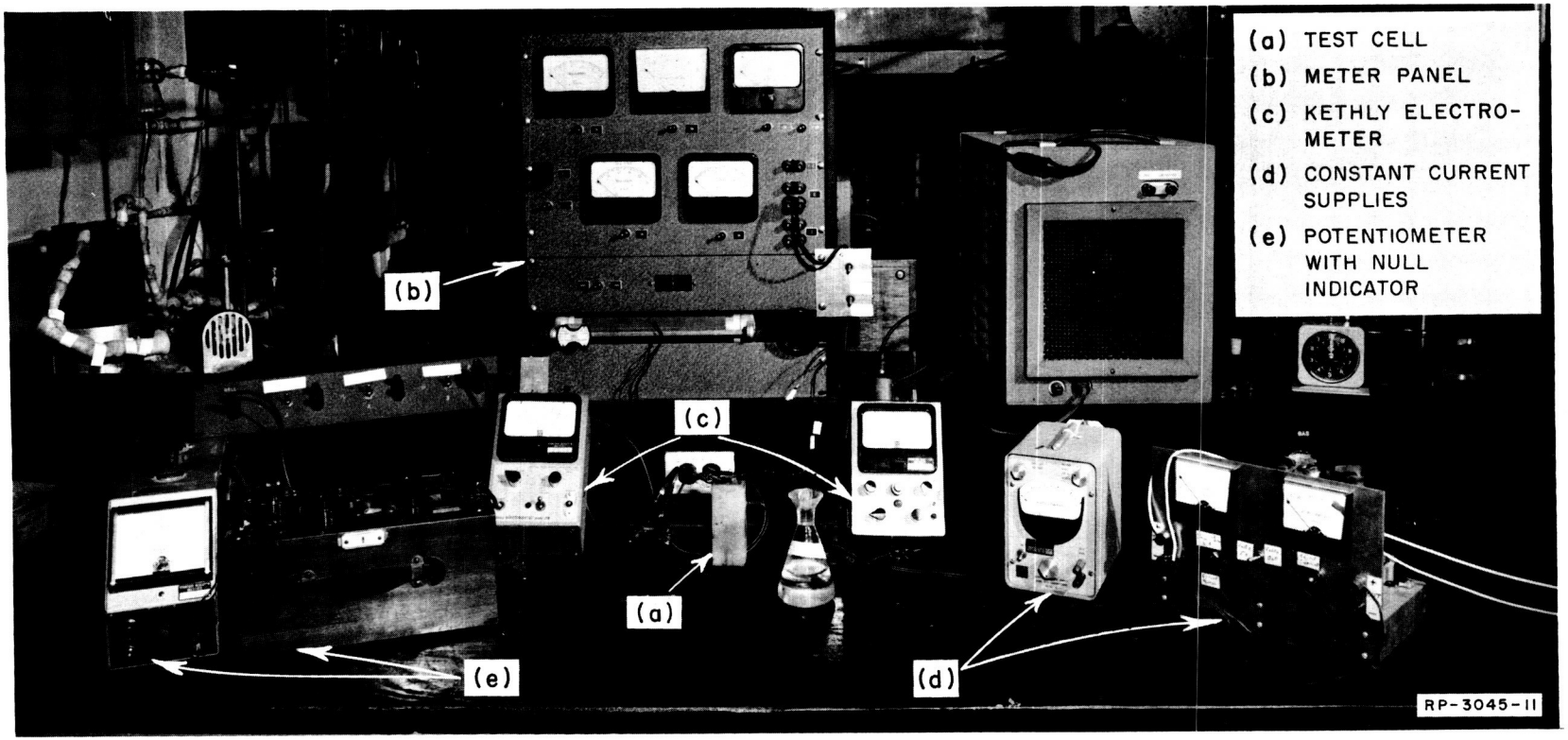

FIG. 11

CELL TESTING EQUIPMENT

The bridge balance point was measured visually with a Tektronix, Type 531, oscilloscope (d).

3. Cell Capacity

The capacity of Halane-containing cells was determined in runs with 2 gram Halane present in the total mixture. For a 10% voltage drop, a capacity of 270 ma-min* was found to be independent of the cell construction, as far as the use of an ion exchange membrane or filter paper for diaphragm was concerned. The cell voltages depended on the cell construction; the higher voltages were obtained at smaller resistances, e.g., 3.30v at 10 ma rate for the lower resistance cells with filter paper. Halane utilization was 0.4% for the 10% voltage drop. These results are recorded in Table VII.

The capacity of $KlCl_4$-containing cells was determined in runs with 2.0 grams $KlCl_4$ present in the total mixture. For a 10% voltage drop, a capacity of 1,050 ma-min was found at 10 ma current, and 1,250 ma-min at 50 ma current. These results are recorded in Table VIII.

F. POLARIZATION STUDIES

Polarization values of both electrodes were evaluated using a cell assembly designed to this end shown in Fig. 12.

Platinum reference electrodes were immersed in the electrolyte contained by pockets in the cell walls. The potential measurements made between the platinum reference electrodes and the main electrodes were a function of the activation and concentration polarization and of resistance polarization from the voltage gradient in solution. Data are given in Appendix B. Cells run with Halane-graphite cathodes, lithium anodes, and propylene carbonate-potassium thiocyanate solution indicate that a large part of the polarization is from resistance of the solution at currents to 100 ma or current densities to (5 ma)/cm^2. The resistance of the cell is high because of the nature of the solvent-electrolyte used.

* Milliampere-minutes.

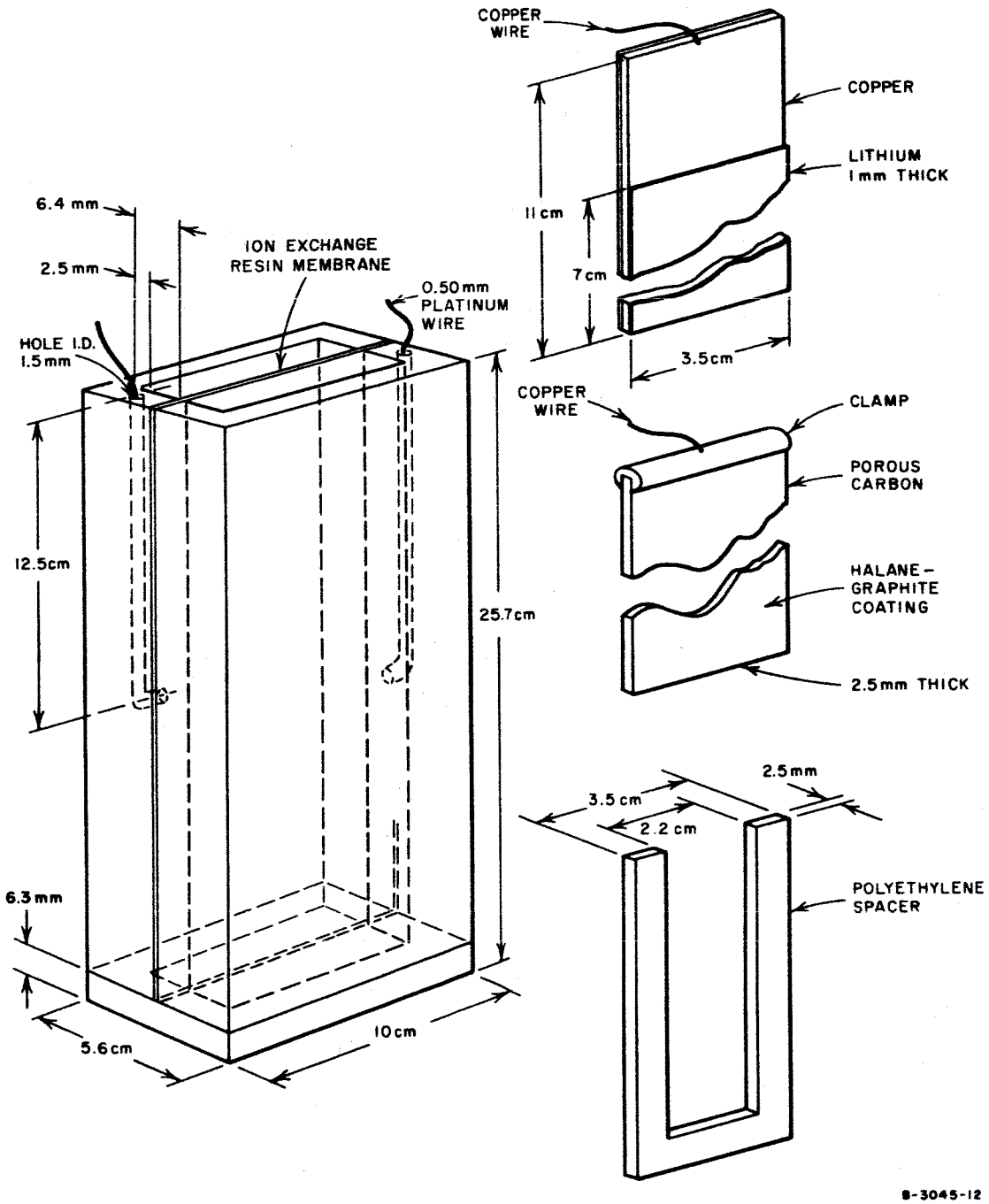

FIG. 12

CELL FOR POLARIZATION STUDIES—TYPE III

The resistivity of the standard potassium thiocyanate-propylene carbonate solution used is 146 ohm-cm. The total resistance of the parallel plate cells closely approximates that calculated by the equation $R = \rho L/A$ where L is the distance between plates and A the area exposed. Calculation of concentration polarization E_e for the cathode by the usual equations

$$\Delta E_c = \frac{nF}{RT} \ln \frac{1 - i}{i_L} \qquad \text{and} \qquad i_L = \frac{nFDC_0}{\delta}$$

shows that E_c is of minor contribution at currents less than 100 ma. For values of $C_0 = 6 \times 10^{-4}$ mole/cc, N = 2 equivalents per mole, $D = 10^{-5}$ cm^2/sec, $\delta = 0.005$ cm, T = 298°K, and i = 100 ma, ΔE_c is 3 mv.

Polarization experiments also indicated that the potential of a platinum reference electrode versus the lithium anode remained practically unchanged, while the terminal anode-cathode voltage dropped to zero. This indicated that a serious limitation of the cell is in the Halane cathode. It is believed that the difficulties with the Halane electrode are caused by the formation of a gaseous (chlorine) or solid (parathiocyanogen) resistive layer which interferes with the contact between the carbon grains and the active material.

Preliminary experiments to determine the site of the high "internal resistance" were not successful, due to inconsistent and rapidly changing results. Resistance-capacitance measurements were also erratic because of chemical changes in cells. These were carried out by potential-time measurements using a reference electrode on both sides of the separator and an oscilloscope with 10^{-6} second cut-off times.

G. KCNS PURIFICATION AND ANALYSIS

One hundred twenty grams of commercial KCNS were placed in a clean container and heated at 70°C in an evacuated oven (1 mm pressure) until dry. After about four hours the salt was removed, ground in a mortar and pestle, and reheated in the vacuum oven for twenty-four hours. The salt was then removed and placed in a vacuum desiccator until ready to be placed in solution for cell measurements.

Solutions KCNS in propylene carbonate were analyzed by titrating with a standard silver nitrate solution.

IV DISCUSSION

Lithium anodes of sufficiently low polarizability and compatibility are possible in nonaqueous electrolytes. The most promising systems are propylene carbonate - KCNS, or Et_4NBr as electrolytes with cathodes of Halane or $KICl_4$.

Total cell voltages of 3.9 have been obtained, which surpass those of most other battery systems.

A major part of this work was concerned with determining the conductivity of nonaqueous electrolytes which were inert to lithium. A suitable system was propylene carbonate with near-saturated solutions of KCNS or Et_4NBr. Specific conductivities of the order of $K = 7 \times 10^{-3}\ \Omega^{-1}\ cm^{-1}$ at 25°C were obtained. This is about one hundred times more resistant than sulfuric battery acid ($0.7\ \Omega^{-1}\ cm^{-1}$. This shows that one of the limiting factors of the lithium battery capacity was the high resistance of the electrolyte.

Current densities of 20-25 ma/cm^2 were obtained with cell experiments using lithium anodes, Halane cathode, and propylene carbonate-KCNS-H_2O mixtures. Typical runs are shown in Appendix B, experiments 35-37. Similar current densities were obtained with lithium anodes, $KICl_4$ cathodes, and a propylene carbonate-KCNS electrolyte, as shown by experiments 71, 73, and 75. The power-to-weight ratio was approximately doubled by the use of bipolar cells in which double cell construction gave similar current densities but at higher voltages. These experiments (102 and 103) indicated that another major limitation of the present battery system is in the high polarizability under current drains. Future work would definitely have to concentrate on this side of the cell system.

A membrane of suitably low resistance and good compatibility with the system is also lacking, although "Pormax" showed good results.

V FUTURE WORK

Future studies should concentrate on improvement of the cathode reaction. Any further development of this battery will depend primarily on what improvements can be made in the proper selection of cathode material to give lower polarizability and higher charge-to-weight ratio. This means work must focus on examination of different cathode materials, organic and inorganic. Further systems which are suggested for consideration as cathode materials are Li_2O_2, percarbonates, perborates, perfluorates, persulfates, perphosphates, potassium chlorate, iodoform, CI_4, T-chloramine, N-chloramine, and organic acids.

The question of the location of the main part of the resistance indicated by the D.C. potential measurements should be studied by A.C. impedance measurements with frequencies up to 40 kc/sec.

Attempts to improve the electrolyte conductivity should be made to form solvent-additive complexes. These may be found in components such as $AlCl_3$ and fluorides.

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APPENDIX A

MATERIALS AND PROCEDURES FOR CATHODE "CAKE," $KICl_4$, AND Cu_2S PREPARATION

Appendix A

MATERIALS AND PROCEDURES FOR CATHODE "CAKE," $KICl_4$, AND Cu_2S PREPARATION

Cathode Cakes

No. 1

2 gm Halane

0.8 gm powdered graphite

0.2 gm Flexbond resin (polyvinyl alcohol)

Materials were wet with small amount of acetone, spread between polyethylene form, air dried 30 minutes, and placed in 90°C oven for 1 hour. Product showed swelling and, although strong, had a very irregular surface.

No. 2

4 gm Halane

1.6 gm powdered graphite

0.2 gm Flexbond resin (PVA)

Materials were mixed together, placed in 1-inch circular die, and pressed at a pressure of 6,000 pounds. A strong compact cake, 5/16 x 1 inch was formed.

No. 3

24 gm Halane

9.6 gm powdered graphite

Material was mixed and placed in a rectangular die. Mix and die were placed in Baldwin machine which applied a total pressure of 72,000 pounds. A smooth, compact cake 3.5 mm thick by 15 cm x 5 cm was formed.

No. 4a

16 gm Halane

8 gm KBr

8.0 gm graphite

Materials were mixed and pressed in die at 85,000 pounds pressure. A very poor cake which broke very easily was formed.

No. 4b

16 gm Halane

8 gm KBr (ground to a fine powder)

9.6 gm graphite

Materials were mixed together with 10 drops raw propylene carbonate. They were ground, placed in die, and pressed at 75,000 pounds. This cake held together well and was usable as a cathode in cell.

Cathode Cakes

No. 5

16 gm graphite

16 gm CuO

0.8 gm Flexbond resin (PVA)

10 ml acetone (to dissolve resin)

Components were ground together using a mortar and pestle. After grinding, material was placed in a die, spread evenly, air dried 18 hours, and pressed at 80,000 pounds pressure. A fairly good cake resulted.

$KICl_4$

KCl 7.5 gm

I_2 12.7 gm

Cl_2 7.1 gm

KCl was mixed with a little water to which I_2 was added and heated to 50°-60°C in a water bath. Cl_2 bubbled in slowly as orange crystals formed. Crystals of $KICl_4$ were dried with suction and stored in a desiccator.

Cu_2S

Cu wire 6.4 gm

S 4.0 gm

Cu and S were heated in a partially closed crucible until excess S burned off. Cu_2S was then crushed and powdered.

APPENDIX B - PARTS 1 AND 2

SUMMARY OF RESULTS OF CELL EXPERIMENTS

APPENDIX B - PART 1

SUMMARY OF RESULTS OF CELL EXPERIMENTS

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
1	Aluminum backing plates	Graphite-Halane (cast bar) 20 gm-Halane, 10 gm-graphite	KCNS (10 gm / 50 ml solvent)	Propylene carbonate	19.0	3.0	2.5	1.32×10^{-4}	700	--	Cellophane	I	Cathode has orange coating on surface. Anode - clean and shiny.
2			KCNS saturated	Acetonitrile	19.0	2.35	5.5	2.89×10^{-4}	540	--			Anode and cathode have light yellow coating - some attack on cathode.
3				Dimethyl sulfoxide	19.0	3.40	20.0	10.5×10^{-4}	148	--			Anodes clean. Solution slightly yellow. Very strong sulfur odor.
4				Dimethyl formamide	19.0	2.70	3.0	1.57×10^{-4}	>690	--			As No. 3.
5				γ Butyrolactone	19.0	3.50	50.0	26.3×10^{-4}	64	--			Anodes clean. Red-brown liquid in cathode side.
6	Brass screen Li rolled on	30 gm-Halane, 10 gm-graphite	KCNS (10 gm / 50 ml solvent)	Propylene carbonate	19.0	4.00	0.78	4.1×10^{-5}	--	--	Polyvinyl alcohol		Solution yellow-orange coating on diaphragm and cathode. Anodes OK - except white film above liquid level. Polyvinyl alcohol dissolves to a gummy mass.
7	Aluminum backing plates	20 gm-Halane, 10 gm-graphite	KCNS saturated	γ Butyrolactone	19.0	3.42	8.0	4.2×10^{-4}	9570	--	Cellophane-soaked in methanol for two weeks	I	Anodes - white frothy material at liquid level. Cathode - yellow to red coating on surface. Solution pink in color.
8	Brass screen Li rolled on	30 gm-Halane, 10 gm-graphite	KCNS (10 gm / 50 ml solvent)	Propylene carbonate	19.0	3.30	0.70	3.7×10^{-5}	>100	--	Cellophane	I	Similar to No. 7.
9					19.0	3.45	4.6	2.42×10^{-4}	344	--	Filter paper*	I	Dark red solution. Li good - alloying of Li to Al at contact points. Anode position 2.
10					24.0	2.40	0.8	3.3×10^{-5}	400	--	Filter paper*	I	Solution a straw color. Anodes in good condition. Anode position 3.
11		Brass screen dipped into molten Halane-graphite	KCNS (9.81 gm / 50 ml solvent)		31.50	2.40	10.0	3.18×10^{-4}	20	--	3 sheets filter paper	II	Clips in liquid. Li anode in good condition.
12		Graphite Halane cast bar	KCNS (10 gm / 50 ml solvent)		19.00	3.1	2.1	1.10×10^{-4}	1450	--		I	Solution yellow. Anodes in good condition. Anode position 3.
13	Cu sheet brass screen Li rolled on	Graphite Halane cast bar	KCNS (9.4 gm / 50 ml solvent)		15.0	2.1	12.5	8.33×10^{-4}	30 42 in 36 min.	--		II	Solution yellow. Anodes in good condition.
14	Cu sheet brass screen Li rolled on	Cu sheet graphite Halane coated on brass screen	KCNS (9.85 gm / 50 ml solvent)		15.70	1.7	17.0	10.8×10^{-4}	--	--		II	Solution yellow. H_2O added (5 ml) increased current to 40 ma - heavy gassing.
15			KCNS (9.48 gm / 50 ml solvent)		24.0	1.9	40.0	16.66×10^{-4}	15	--	3 sheets filter paper*	II	Black film on copper backing plate. Solution yellow. Papers yellow-orange in color. Copper beneath black film - bright and shiny.
16	Al plate perforated with 5/32 in. holes. Li pressed into perforations	Al plate brass screen	KCNS (9.48 gm / 50 ml solvent)		26.0	2.30	4.3	1.72×10^{-4}	--	--	3 sheets filter paper*	II	Solution yellow. Anodes - good. Paper - orange color.

* #42 Whatman Filter Paper

(dist.) - Distilled

ml - milliliter

APPENDIX B - PART 1 (Continued)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
17	Al plate perforated with 5/32 in. holes. Li pressed into perforations	Al plate brass brass screen	KCNS 9.48 gm / 30 ml solvent	Propylene carbonate	26.25	3.10	3.1	1.18×10^{-4}	--	--	Cellophane	II	Solution yellow-orange. Anode - good.
18	As above except Cu and Brass parts gold plated		KCNS 9.20 gm / 30 ml solvent		26.40	2.20	11.0	4.13×10^{-4}	--	--	Cellophane	II	Solution dark yellow. Anodes - good. Black film on surface of gold - easily removed.
19	Graphite bar - Li	Graphite bar coated with graphite-Halane	KCNS 9.20 gm / 30 ml solvent		16.26	3.20	67.0	41.1×10^{-4}	Approx. 10	--	3 sheets filter paper*	II	Anode - good. Absorption of solution by carbon so that only 10 ml (of 30) remained at end of run.
20	Graphite bar - Li	C-60 porous carbon graphite 5 Halane 30 poured over surface	KCNS 9.20 gm / 50 ml solvent		24.0	3.25	200	83.3×10^{-4}	Approx. 9	--	3 sheets filter paper*	II	Graphite back plate for Li was chewed away beneath Li. Anode good.
21	Al back plate Li pressed into perforations	C-60 porous carbon graphite 5 Halane 30 poured over surface	KCNS 9.20 gm / 50 ml solvent		16.25	3.42	110	67.4×10^{-4}	30	--	6 sheets filter paper*	II	Solution clear with an orange precipitate. Sheet of paper at Halane end orange. Sheet of paper at Li end white. Anode gray.
22	Al back plate copper probe between 3rd and 4th paper	C-60 porous carbon graphite 5 Halane 30 poured over surface	KCNS 9.82 gm / 50 ml solvent		16.25	3.35	105	64.4×10^{-4}	--	--	6 sheets filter paper*	II	Copper wire probe has a black easily removed film on it. Solution yellow.
23	As No. 22 except probe is between 1st and 2nd paper at Li end	C-60 porous carbon. graphite 5 gm Halane 30 gm poured over surface	KCNS 9.82 gm / 50 ml solvent		20.48	3.25	130	63.4×10^{-4}	10	--	6 sheets filter paper*	II	Cell leaked.
24	As No. 23 except Cu wire gold plated	C-60 porous carbon. graphite 5 gm Halane 30 gm poured over surface	KCNS .10 gm / 50 ml solvent		18.0	2.8	5.0	2.7×10^{-4}	--	--		II	Solution yellow. Gold plated wire - bright.
25	As No. 23	C-60 porous carbon graphite 5 gm Halane 30 gm poured over surface	KCNS 10 gm / 50 ml solvent		22.4	3.46	110	49.1×10^{-4}	--	--		II	
26	Al backing plate Li. Au plated Cu wire. Probe between 1st and 2nd papers at Li end	C-60 carbon-graphite-Halane coated	Li Br 5 gm / 50 ml solvent	(Saturated milky)	20.80	3.62	64	30.7×10^{-4}	50	--	6 sheets filter paper*	II	Solution yellow. Papers yellow.
27	As No. 26	C-60 carbon-graphite-Halane coated	NH_4Cl 10 gm / 50 ml solvent	Propylene carbonate	19.50	4.0	6	3.14×10^{-4}	530 initial 550 in 11.6 min.	--		II	Anodes good. Solution pale yellow. All papers white.

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter

APPENDIX B - PART 1 (Continued)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
28	As No. 26	C-60 carbon-graphite-MnO_2 NH_4Cl-glue, H_2O mixed and dried	KCNS (10 gm / 50 ml solvent)	Propylene carbonate	20.0	3.4	100	50 x 10^{-4}	16	--		II	Solution clear. Paper white. Anode good.
29		C-60 carbon (air cell)	KCNS (10 gm / 50 ml solvent)	Propylene carbonate	18.60	3.2	64	34.4 x 10^{-4}	13	--		II	Solution yellow. Foamy. Papers white.
30		C-60 carbon-graphite-Halane coated	KNO_3 10 gm (40% dissolved)		19.50	3.85	9.5	4.9 x 10^{-4}	380	--		II	
31			Li Br - 5 gm Et_4N Br - 5 gm in 100 ml	Dimethyl sulfoxide	16.10	2.2	46	1.4 x 10^{-4}	>15	--	6 sheets Kraft paper	II	This cell prepared in laboratory. Li turned gray by the time the cell was assembled. Not much Halane on graphite.
32	Al backing plate Li strips		Li Br - 5 gm Et_4N Br - 5 gm in 100 ml		18.0	3.30	170	94.4 x 10^{-4}	--	--	6 sheets filter paper*		Liquid brown in color - carbon in suspension. All papers yellow.
33	Cu sheet perforated Li strips		NH_4Cl - 5 gm LiCl - 5 gm in 50 ml	Propylene carbonate	19.5	3.55	13.0	6.67 x 10^{-4}	23,600	--			Solution dark green. All papers green in color. Heavy attack of Halane-graphite.
34			NH_4Cl - 5 gm LiCl - 5 gm in 50 ml	Propylene carbonate 48 ml H_2O - 2 ml (dist.)	19.60	3.70	6.8	3.5 x 10^{-4}	55,000	--			Solution clear. Papers white at finish of run.
35			KCNS (10 gm / 50 ml solvent)	Propylene carbonate 40 ml H_2O - 10 ml (dist.)	19.5	3.2	500 dropped to 200	102.5 x 10^{-4}	--	--			Solution dirty yellow-green color. Papers yellow at Li end. Red-brown at Halane.
36				Propylene carbonate 45 ml H_2O - 5 ml (dist.)	21.0	3.5	230 dropped to 160 in 5 min	109.5 x 10^{-4}	--	--		II	Paper yellow at Li end. Orange at Halane end.
37				Propylene carbonate 50 ml H_2O - 2 ml (dist.)	22.5	2.90	380 to 310 in few sec. 115 in 2 min.	137.8 x 10^{-4}	Approx. 30	--	6 sheets filter paper*	II	Solution dark mustard color. Papers yellow. Very heavy yellow coating on carbon.
38				Propylene carbonate	19.04	3.20	110 dropped to 31 in 47 min	57.8 x 10^{-4}	Approx. 30	--	6 sheets filter paper*		Solution slightly yellow. Papers slightly yellow at Halane end. White at Li end.
39					7.2	1.0	11	15.2 x 10^{-4}	--	--	Pyroxylin film		1 gm Halane placed in carbon compartment. Solution red-brown. Diaphragm dissolved.
40	Molten Li dipped onto Cu sheet				15.0	3.50	120	80 x 10^{-4}	--	--	6 sheets filter paper*		Solution yellow. Papers orange-red at Halane, decreasing to slight yellow at Li end.
41	Molten Li dipped onto Cu sheet			Propylene carbonate H_2O - 2 ml (dist.) and same Propylene carbonate	15.0	3.20	200 dropped to 60 in 23 min	133.3 x 10^{-4}	--	--	6 sheets filter paper*		Solution light yellow. Papers yellow at Halane end. White at Li end.
42	Cu - Li strips			Propylene carbonate	15.0	3.40	0.20	1.3 x 10^{-5}	--	--	Cellophane (DuPont)	III	Cell leaked. Cell cut in half and cemented together with rubber cement. Cellophane separated between halves.

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter

APPENDIX B - PART 1 (Continued)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
43	As No. 42	As No. 42	KCNS 10 gm 50 ml solvent	Propylene carbonate	25.20	3.55	360 to 100 in 10 min.	14.3 x 10^{-4}	30	--	1 sheet filter paper*	III	Solution yellow in Halane side. Solution clear in Li side.
44					25.90	2.50 2.70 38 min.	0.4 to 42.5 in 21.3 min.	16.4 x 10^{-4}	>700	--	"Nalco" ion exchange resin (0.003 in. thickness)		Solution yellow in both chambers. Cracked membrane.
45					23.5	3.3	170 80 in 20 min.	72.3 x 10^{-4}	--	--	Fiberglass ion exchange resin-green		Solution in Halane side the color of motor oil. Membrane surface rough.
46					23.8	3.3	200 80 in 7.28 min.	84.1 x 10^{-4}	--	--			Li side solution yellow. Halane side color of motor oil. Some leakage of solution from cell.
47					23.45	3.1	150 48 in 12 min.	63.8 x 10^{-4}	--	--			Solution in Halane side orange in color. Li side light orange.
48					23.28	3.4	55- rapid drop	23.6 x 10^{-4}	28.5	--			Solution in Halane side red-brown color. Li side lighter in color.
49					22.75	3.30	40	17.5 x 10^{-4}	29 (31.5°C)	--	Fiberglass ion exchange resin-green		Solution in Halane side dark red. Li side light yellow. Yellow-red film on Halane surface.
50			Saturated with argon prior to run		23.45	3.10	40	17.02 x 10^{-4}	--	--			1 gm Halane and 1 gm KCNS added to cathode side. Membrane is dark red in color. Solution in Halane side dark red. Li side light yellow.
51	Li electro-plates on Cu sheet (satu-rated Li Cl - propylene carbonate bath)		$\left(\frac{\text{KCNS } 10\text{ gm}}{50\text{ ml solvent}}\right)$		20.0	3.40	120- drop to 65 in 3 min.	60.0 x 10^{-4}	--	--			Solution in Halane side has a large quantity of loose carbon. Li side yellow in color. Membrane dark red. Graphite-Halane orange coating.
66	Li dipped	C-60 carbon impregnated with Halane dissolved in acetone			23.2	2.30	120	51.7 x 10^{-4}	814	26			Solution yellow-orange. Carbon broke into many pieces during removal. Li good.
67		1 gm Halane			19.2	2.15	165	86.1 x 10^{-4}	8.2	2.0			Halane-carbon broke on removal - chewed up surface. Solution yellow.
68		1 gm Halane			16.32	2.90	110	67.4 x 10^{-4}	32.6	4.1			Solution red. Copper corroded - green copper salt compound.
69		1.58 Halane			20.9	3.35	190	90.9 x 10^{-4}	12.5	2.2			Halane-carbon broken 3 cm from top connector.
70		C-60 carbon O_2 (in air)			22.4	0.92	70	31.2 x 10^{-4}	7.5	4.1 (wire) 8.1 (carbon)	1 sheet filter paper* between spacers		Carbon broken - poor run.
71		2 gm $KICl_4$ on C-60 carbon			25.6	3.10	470	183.6 x 10^{-4}	6.7 5.0 6.7	3.10 1.2	1 sheet filter paper*		Solution brown. Li light tan color. Carbon broke one inch from top connection when being removed. Orange precipitate in propylene carbonate.
72		1.58 gm Halane			29.75	2.90	270	90.6 x 10^{-4}	7.1 8.0	2.1 1.7	1 sheet filter paper*		Solution deep red color. Li coated with a red film. Black sludge in bottom of cell. No yellow precipitate.
73		1 gm $KICl_4$ 0.8 gm graphite wet with acetone			17.6	3.20	300	17.04 x 10^{-4}	9.6	--	1 sheet filter paper* at carbon face		Solution deep red color. Li coated with a red film. Black sludge in bottom of cell. No yellow precipitate.

* #42 Whatman Filter Paper

(dist.) - Distilled

ml - milliliter

APPENDIX B - PART 1 (Continued)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
74	Li dipped onto Cu in dry box	2 gm Halane 0.8 gm graphite powder. Mix with acetone on prewet carbon	35 ml of No. 7 cut propylene carbonate plus 7 gm KCNS (dried) saturated solution	Propylene carbonate	17.6	2.58	100	57.1×10^{-4}	13.6	17.6	1 sheet filter paper*	II	Heavy orange precipitate. Li coated, also.
75	↓	1 gm ethylene diamine $KICl_4$ 0.8 gm graphite mix with acetone spread on C-60 carbon	KCNS (10 gm / 30 ml solvent)	30 ml used	15.4	2.45	180 dropped to 150	1116.8×10^{-4}	2.7	12.0	2 sheets filter paper* against carbon	II	Solvent pale yellow. Paper orange. No precipitate.
77		2 gm Halane 0.8 gm graphite as above	↓	25 ml used	16.8	3.60	Time run at 50 ma		--	--	2 sheets filter paper* 1 spacer and 1 spacer wedge behind Li electrode	II	Paper orange colored. Black coating on paper next to carbon. Solution orange.
78		2 gm Halane 0.8 gm graphite powder		25 ml used	16	3.5	10		--	1 min. 20	2 sheets filter paper*	II	Yellow precipitate. Red liquid.
79		2 gm Halane 0.8 gm graphite powder		25 ml used	24.75	3.40	50		--	--	Green ion-exchange resin (soaked in propylene carbonate KCNS overnight)	III	Electrodes placed against membrane on opposite sides. Spacers placed behind electrodes to wedge them firmly against membrane. Yellow-orange precipitate and color on carbon side. Clean on Li side.
80		2 gm Halane 0.8 gm graphite powder		30 ml used	22.4	3.60	10		16.2 at end of run	--	Green ion-exchange resin (soaked in propylene carbonate KCNS overnight)	III	Li side solution clear. Gray coating on carbon Halane.
81		2 gm $KICl_4$ 0.8 gm graphite wet with acetone and spread on C-60 carbon		30 ml used	21.76	3.62	100		7	--	1 sheet filter paper* and 1 spacer	II	Orange precipitate on back of carbon. Liquid deep red in color.
82		2 gm $KICl_4$ 0.8 gm graphite (acetone wet and spread on carbon)		30 ml used	17.55	3.55	10		5.82	--	1 sheet filter paper* and 1 spacer between electrodes	II	Li dark gray.
83		2 gm Halane 0.8 gm graphite wet with acetone and spread on C-60 carbon		30 ml used	21.45	3.40	72	33.5×10^{-4}	40 at finish	--	Green ion exchange resin	III	Cathode has heavy orange coating. Li good. Liquid orange on cathode side, clear on Li side.
84	↓	2 gm $KICl_4$ 0.8 gm graphite	↓	30 ml used	22.4	3.40	110	49.2×10^{-4}	28.2 28.8 at end	--	Green ion exchange	III	

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter

APPENDIX B - PART 1 (Continued)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
85	Li dipped on Cu in dry box	2 gm S 1 gm KCN 1 gm graphite wet with acetone spread on C-60 carbon	KCNS 10 gm / 50 ml solvent	Propylene carbonate	17.6	2.80	250	142 x 10^{-4}	12.3 at finish	--	1 sheet filter paper* against cathode and 1 spacer between electrodes	II	Coating sloughed off cathode into bottom of cell solution otherwise colorless.
86		2 gm $KICl_4$		30 ml used	20.8	3.45	50	24.0 x 10^{-4}	7.42 21 at finish	--	Green ion exchange resin	III	Both electrodes against membrane spacers behind them. Li coating red - obvious leakage. Li side deep red and clear. Carbon side deep red with orange precipitate.
87		0.8 gm graphite wet lightly with propylene carbonate and spread on C-60 carbon (prewet with propylene carbonate)			19.2	3.10	180	93.8 x 10^{-4}	6.25 8.4 at finish	--	1 sheet filter paper* against graphite, 1 spacer between electrodes	II	Solution colorless except for graphite in liquid. Solution yellow color on Li.
88		2 gm Di** 0.8 gm graphite wet lightly with propylene carbonate and spread on C-60 carbon			20	3.50	200	100 x 10^{-4}	18.3 at finish	--	1 sheet filter paper* against Di,** 1 spacer between electrodes	II	Caked orange precipitate on side away from carbon. Solution pale orange. Heavy orange precipitate.
89		2 gm $KICl_4$ 0.8 gm graphite wet with propylene carbonate and spread on C-60 carbon			20	3.8	445	222.5 x 10^{-4}	7.15 18.5 at finish	--	1 sheet filter paper* against cathode, 1 spacer between electrodes	II	Paper red-orange in color. Solution deep red in color.
90		2 gm S 1 gm KCN 0.8 gm graphite 0.1 gm PVA resin-acetone wet with propylene carbonate on C-60 carbon baked 1 hr at 90°C			26.4	2.78	230	87 x 10^{-4}	8 11.6 at finish	--	4 sheets filter paper*	II	Cathode coating facing away from Li anode (rerun - No. 91).
91		2 gm S 1 gm KCN 0.8 gm graphite 0.1 gm PVA resin-acetone wet with propylene carbonate on C-60 carbon baked 1 hr at 90°C			24.09	2.65	280	116 x 10^{-4}	6 10.6 at finish	--	4 sheets filter paper*	II	Papers white. Solution very pale yellow. Very little loose graphite in solution. Coating easily removed from carbon surface.
92		2 gm $KICl_4$ 0.8 gm graphite wet with propylene carbonate spread on C-60 carbon		25-30 ml used	22.11	3.39	10	4.52 x 10^{-4}	38.8 RT-27°C	--	Green ion exchange resin 1 sheet at cathode	III	Li surface yellow. Paper has large amount precipitate. Orange-red. Back of carbon yellow-orange precipitate. Solution dark red.

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter ** Di - Dibromantin

APPENDIX B - PART 1 (Continued)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
93	Li dipped on Cu in dry box	1.0 gm KCN 0.8 gm graphite wet with acetone 0.1 gm PVA resin (Flexbond) spread on C-60 carbon and baked 1 hr at 90°C	KCNS / 10 gm / 50 ml solvent	Propylene carbonate 30 ml used	28.0	2.70	200	71.4 x 10^{-4}	9.2 12.60	--	1 sheet filter paper* on carbon	II	Paper still white at finish of run. Li clean and no yellow film. KCN coating still on carbon but easily removed. Solution - water clear.
94		2 gm sulfur 1 gm KCN 0.8 gm graphite 0.1 gm PVA resin acetone spread on C-60 carbon baked 1 hr at 90°C			20	2.70	50	25.0 x 10^{-4}	9.85 9.50	--	1 sheet filter paper*	II	Li clean and shiny. Coating still intact on carbon. Solution light yellow in color.
95		As No. 92 except electrolyte solution on both sides of membrane			24.75	3.40	10	4.04 x 10^{-4}	2.5 70 RT-28°C	--	Green ion exchange resin 1 sheet filter paper* at cathode	III	Li has yellow coating. Cathode red-orange color on paper. Solution slightly yellow in Li side. Solution slightly red in carbon side.
96		2 gm Halane 1 gm graphite 1 gm KBr ground in mortar and pestle placed in 1-in. die and pressed under 6,000 lbs. A slug 1 in. x 5 mm was formed. Graphite (aldag) coated Cu.			17.5	3.2	200	114 x 10^{-4}	--	--	1 sheet filter paper*	II	Yellow precipitate. Red liquid. Button soft and falling apart.
97		2 gm sulfur 1 gm KCN 0.8 gm graphite 0.1 gm PVA resin (acetone) on C-60 carbon-dry 1 hr at 90°C			17.5	2.82	10	5.71 x 10^{-4}	8.4 8.7	--	1 sheet filter paper*	II	Li clean. Coating on carbon still good. Liquid very pale yellow.
98		Cake No. 3			18.5	2.30	200	108 x 10^{-4}	4.6 5.8	--	4 sheets filter paper*	III	Liquid dark red. Copper showing through graphite coating - corrosion of Cu.
99		Cake No. 3		25 ml used	20.75	3.10	250	120.5 x 10^{-4}	8.8 8.4	--	4 sheets filter paper*	III	Faint gray coating on Li. First paper at cake - orange-red. Fourth paper faint yellow. Graphite coating in good condition after run. Orange-red coating on Halane cake.
100		24 gm Halane 9.6 gm graphite pressed into cake form in mold. A rectangular piece cut out to fit into polyethylene spacer cake No. 3		25-30 ml used	18.75	3.0	260 drop to 140 immed.	138.2 x 10^{-4}	6.0 14.92 (finish)	--	4 sheets filter paper*	III	1 sheet Cu coated with "Aldag" No. 154. 1 coat baked 1 hr at 100°C. Green coating over Aldag film where cake was in contact. Filter papers orange in color. Yellow coating on cake. Cake itself soft and mushy. Warm to touch.

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
101	Li dipped on Cu in dry box	Cake No. 4B 16 gm Halane 8 gm KBr 9.6 gm graphite	KCNS 10 gm / 50 ml solvent	Propylene carbonate 25-30 ml used	19.2	3.15	240	125 x 10^{-4}	11.1 13.6 (finish)	--	4 sheets filter paper*	III	End sheet of Cu given two coats No. 154 Aldag - 30 min. bake between coats at 100°C - between first and second coat. Brown coat on graphite (Aldag) when rinsed. Blue-black coating on surface. Paper against cake yellow. Fourth paper white. Cake No. 4 in good condition, slightly yellow color on surface. Solution orange-red in color.
102	↓	Cake No. 4B	↓	↓	29.0	7.1	350	120.5 x 10^{-4}	38 (finish)	--	2 sheets filter paper* in each compartment	IV	(Double cell.) Li No. 1 yellowish tinge. Paper yellow-reddish liquid against Li face. Cake No. 1 stuck to paper and soft. Li No. 2 reddish liquid against Li face paper yellow. Cake No. 2 soft and stuck to paper.
103		16 gm graphite 16 gm CuO 0.8 gm PVA dissolved in acetone. 8-10 ml added to cake mix			37.25	5.60	160	62.9 x 10^{-4}	51	--	4 sheets filter paper* in each compartment	IV	Cell-double-anode-cathode. Cake No. 1 paper white Li clear. Cake contacted Li due to short paper. Li No. 1 good. Cake No. 2 soft. Cathode copper deposited on Aldag film about 50% of area. Li No. 2 slight darkening of surface.
104		Cake No. 4B			18.0	2.60	3.3	1.83 x 10^{-4}	700	--	Cellophane 2 sheets filter paper* at cathode	III	Green coating on Aldag film. Li - gray coating. Solution clear in Li side. Red-brown in cathode side. Paper orange at cathode face and dark yellow at membrane face. Cathode yellow on back surface, soft and mushy.
105		16 gm CuS 16 gm graphite 0.8 gm PVA resin		↓	35.0	4.10	120	33.3 x 10^{-4}	--	--	3 sheets filter paper*	IV	Cathode No. 1 - Cu deposit on Aldag film. Papers white. Li No. 1 clean and bright. No. 2 Li and No. 2 cathode same as No. 1. Solution clear.
106	Li strip(Cu backplate)	Porous carbon 2 gm Halane 0.8 gm graphite		50/50 ml % DMS propylene carbonate	10.7	3.90	80	74.7 x 10^{-4}	--	--	3 sheets filter paper*	II	Solution dark yellow but no precipitate. Clear. Papers yellow. Li dark.
107	(2) Li strip on bakelite backplate	Porous carbon 2 gm Halane 0.8 gm graphite		Propylene carbonate	20	3.9			--	--	3 sheets filter paper*	II	Solution red-brown. Papers red, all through. Coating adhered to paper when paper removed from cathode.
110	(1) Li strip on bakelite backplate	0.5 gm graphite 4.0 gm $CuBr_2$ 0.2 gm lucite dissolved in ethylene dichloride mix together grind in mortar and pestle, spread on silver screen, bake 1 hr at 80°C			10.16	1.50	40	39.2 x 10^{-4}	--	--	3 sheets filter paper* One as an envelope around cathode	II	Solution a deep cherry red. Paper in contact with cathode dark red. Li clean, light in color.
111	(2) Li strip	Porous carbon 2 gm Halane 0.8 gm graphite			21.36	3.20	180	84.3 x 10^{-4}	--	--	Pormax	III	Membrane good after run, no swelling, dissolving or tearing. Yellow-orange precipitate on cathode side. None on Li side. Solution orange (transparent) in Li side of cell. Li clean and bright. Cathode coated with yellow precipitate. Li side heavy yellow precipitate.
112	(1) Li strip	Silver screen 0.2 gm graphite Silver precipitate metal 0.5 gm Silver chloride 0.5 gm	↓	↓	10.16	2.25	130	128 x 10^{-4}	--	--	3 sheets filter paper*	II	Solution yellow. No precipitate. Li clean. Cathode material still coating screen.

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter

APPENDIX B - PART 1 (Concluded)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Remarks
									Open	Closed			
113	Li strip on bakelite backplate	1 gm Di** 2 gm graphite 0.2 gm KBr ground with 4 ml propylene carbonate in mortar and pestle porous carbon	KCNS (10 gm / 50 ml solvent)	Propylene carbonate 30 ml used	10.16	2.30	140	137.9×10^{-4}	--	--	Pormax	III	Li clean and bright beneath a yellow film. Yellow coating on surface of Di**. Yellow-green solution in anode side. Most of paste had worked off carbon.
114	Li (dip ct) on moly strip (dry box)	2 gm Halane 0.8 gm graphite	↓	↓	20.5	2.98	290	141.5×10^{-4}	--	--	2 sheets filter paper*	II	Solution yellow. Heavy orange precipitate. Papers yellow to orange. Yellow-orange coating on Li and cathode.
115	↓	1.5 gm Ag_2O_2 1.5 gm precipitated silver metal enough propylene carbonate to make a paste on C-60 carbon			14.4	2.65	160 drops rapidly	111.1×10^{-4}	13.5 25 at end of run	--	1 filter bag 2 sheets filter paper*	II	Solution clear. Paper white. Li good. Peroxide silver mix in good condition on carbon surface.
116		2 gm Halane 0.8 gm graphite			11.05	3.28	100	90.5×10^{-4}	34	--	Pormax (heat sealed to form a bag)	II	Pormax bag black in color (dark gray originally). Orange precipitate in bag, also loose graphite. Li good.

* #42 Whatman Filter Paper (dist.) - Distilled ml - milliliter ** Di - Dibromantin

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Half Cell Voltage	Remarks
									Open	Closed				
52	Li dipped on Cu in dry box	10 gm Halane 5 gm acetylene black 0.1 gm glue-(animal) 24 ml acetone spread on C-60 carbon and dried	KCNS (10 gm / 50 ml solvent)	Propylene carbonate 30 ml used	21.6	3.60	80	37.0×10^{-4}	--	--	Fiberglass ion exchange resin - green	III	Li Halane OCV, 2.0 0.3	Copper quite deeply eroded from molten Li.
53		Halane-acetylene black 0.88 gm of coating			23.1	3.20	37	16.0×10^{-4}	64	--		III	Li Halane OCV 3.05 -0.62	Anode side clear. Cathode side orange color.
54		2 gm sulfur 6 gm graphite powder 4.29 gm coating on carbon			22.8	2.45	58.5	25.7×10^{-4}	30	--		III	Li S OCV, 3.15 0.65	
55		1 gm Di** 2 gm graphite			23.2	3.30	410 dropped to 225 in 15.5 min	176.7×10^{-4}	10.2	--	Green ion exchange	III	Li Dibromantin OCV, 2.56 -0.86 2 ma, 2.70 -0.05	Orange solution in dibromantin side. Paper and graphite solution colored red.
56		4 gm graphite powder 4 ml acetone 0.1 gm D-13 Flexbond resin			21.6	1.8	60	27.8×10^{-4}	34.3	--			Li Graphite OCV, -0.0005 -0.0005 2 ma, -0.0003 -0.0003	Solution orange color on Li side and on graphite side.
57		2 gm graphite 2 gm sulfur			20.8	1.10	63	30.3×10^{-4}	2.8 on 55 ma cell 26.5	--			Li S 2 ma, 2.56 1.00	Solution orange on Li side. Clear on sulfur side.
58		2 gm $CuCl_2$ 2 gm graphite powder 0.1 gm D-13 resin acetone. Dried film on C-60 carbon in vac. oven at 900°C - 20 hr.			21.6	2.86	81	37.5×10^{-4}	33.2 cell 2.9 c.c. 80 ma	--			Li $CuCl_2$ 2 ma, 3.20 -0.18	Solution dark red in cathode side, and light red in anode side.
59		2 gm Di** 2 gm graphite powder 0.1 gm D-13 resin acetone			21.6	2.92	80	37.4×10^{-4}	--	--			Li Dibromantin 2 ma, 3.02 -0.12	Solution deep red in graphite side. Yellow to orange in Li side.
60		2 gm CuO (wire) 2 gm graphite powder 0.1 gm D-13 resin			21.6	0.55	110	50.9×10^{-4}	6.3	1.2	2 sheets filter paper*	III	Li CuO OCV, 2.85 1.90 1 ma, 2.65 1.95	Solution colorless. Large amount graphite powder floating in solution.
61		2 gm CuO fine powder			21.6	1.67	170	78.7×10^{-4}	1.1	--	1 sheet filter paper*		Li CuO OCV, 2.45 1.08 1.1 ma, 2.62 1.20	Solution clear.
62		2 gm CuF_2 2 H_2O 2 gm graphite powder 0.1 gm D-13 resin acetone			22.4	2.30	300	134 $\times 10^{-4}$	Total 1.2 4.4	--	2 sheets filter paper*		Li CuF_2 OCV, 2.20 0.4 1.5 ma, 2.52 0.68	Solution red-brown. Cu deposit on graphite front and back.

* #42 Whatman Filter Paper ml - milliliter **Di - Dibromantin c.c. - closed circuit

APPENDIX B - PART 2 (Concluded)

Cell Run No.	Anode Materials	Cathode Materials	Electrolyte	Solvent	Li Area (cm^2)	OCV	Max. I (ma)	CD (amp/cm^2)	Cell Resistance Ω		Membrane	Cell Type	Half Cell Voltage		Remarks
									Open	Closed					
63	Li dipped on Cu in dry box	2 gm CuS 2 gm graphite powder 0.1 gm D-13 resin acetone	KCNS (10 gm / 50 ml solvent)	Propylene carbonate 30 ml used	20.8	1.90	300 decreases rapidly to 240 in 10-15 sec	144.2 x 10^{-4}	4.1	1.3	2 sheets filter paper*	III	Li OCV, 2.42 1.1 ma, 2.30	CuS approx. 0.75	Li good. Solution black-green. Material on bottom of cell. Cathode deteriorated.
64	↓	2 gm Halane 2 gm graphite 2 gm KCNS 0.1 gm D-13 resin acetone	↓	↓	21.0	2.90	350 decreases rapidly to 210 (2 min)	166 x 10^{-4}	7.3	7.2	2 sheets filter paper*	III	Li OCV, 2.92 1.8 ma, 2.92	Halane -0.36 -0.35	Solution yellow-brown. Filter papers yellow. Li good.
65		2 gm Halane 1.5 gm graphite 2 gm KCNS 1 gm KBr 0.1 gm D-13 acetone ground together			18.9	2.85	310 decreases rapidly	164 x 10^{-4}	12.40	2.58	2 sheets filter paper*	III	Li OCV, 2.88 2 ma, 2.88	Halane -0.23 -0.08	Solution red-brown. Papers red.

* #42 Whatman Filter Paper ml - milliliter Di - dibromantin c.c. - closed circuit